

# X-ray Optical Components for Synchrotron Radiation

Dennis M. Mills  
Deputy Associate Laboratory Director  
Advanced Photon Source

National School for Neutron and X-ray Scattering  
August 2012

# Outline

## Outline of Presentation

1. Why Do We Need Optics?
2. X-ray Mirrors (Reflective Optics)
3. Perfect Crystal X-ray Optics (Diffractive Optics)
4. Focusing Optics (Reflective, Diffractive and Refractive)

# Why Do We Need Optics

- Control the energy (E) and bandwidth ( $\Delta E$ ) of the beam.
  - Monochromatic beam ( $\Delta E = 1\text{-}2\text{ eV @ } 10\text{ keV}$ ;  $\Delta E/E = 10^{-4}$  )
  - Polychromatic ( $\Delta E = 1\text{-}2\text{ keV @ } 10\text{ keV}$ ;  $\Delta E/E = 10^{-1}$  )
  - High-resolution beams ( $\Delta E = \text{a few milli-eV @ } 10\text{ keV}$ ;  $\Delta E/E = 10^{-7}$  )
- Control the size/divergence of the beam (often related).
  - Micro or nano beams (spot sizes microns to 10's of nanometers)
  - Highly collimated beams
- Control the polarization of the beam.
  - Linear
  - Circular

# Reflective Optics: the Dielectric Constant at X-ray Wavelengths

- Since the reflective properties of a material depends on the index of refraction, we need know this at x-ray energies/frequencies. It pretty straight forward to calculate the dielectric constant for a material using the Drude Theory (simple free electron model) – See Appendix 1:

$$\kappa = 1 + 4\pi(P/E) = 1 + 4\pi (e^2/m)n_e [1/(\omega_o^2 - \omega^2)]$$

where  $m$  is the mass of the electron,  $e$  is the charge of the electron,  $n_e$  is the electron density,  $\omega$  the frequency of the x-rays, and  $\omega_o$  some natural frequency of the electrons in the solid.

- In the Drude model  $\omega_o$  the frequency of the collective oscillations of the electron gas around the positive ion background or the so-called plasma frequency and equal to:

$$\omega_o = [4\pi n_e e^2/m]^{1/2}$$



# Index of Refraction at X-ray Wavelengths

- For Si,  $n_e = 7 \times 10^{23} \text{ e/cm}^3$  and so the plasma frequency is:

$$\omega_o = 5 \times 10^{16}/\text{sec}$$

- For a 1 Å x-ray, the angular frequency,  $\omega = [2\pi c/\lambda]$ , is  $2 \times 10^{19}/\text{sec}$  which is  $\gg \omega_o$  and so we can write:

$$\kappa = 1 + 4\pi (e^2/m)n_e [1/(\omega_o^2 - \omega^2)] \approx 1 - 4\pi (e^2/m)n_e [1/(\omega^2)]$$

- The index of refraction,  $n$ , is just the square-root of the dielectric constant and can be written as:

$$n = \kappa^{1/2} = [1 - 4\pi((e^2/mc^2) c^2 n_e (\lambda/2\pi c)^2)]^{1/2}$$

$$n = \kappa^{1/2} = [1 - (n_e(e^2/mc^2) \lambda^2/\pi)]^{1/2} \approx 1 - 1/(n_e r_e/2\pi) \lambda^2$$

where  $r_e = (e^2/mc^2)$  is the classical radius of the electron ( $2.82 \times 10^{-13} \text{ cm}$ ).

# Index of Refraction, $n < 1$

- This is often written as:

$$n = 1 - \delta$$

where  $\delta = (n_e r_e / 2\pi) \lambda^2$ .

- When you plug in the numbers for the real part of the index of refraction you get:

$$\delta = 10^{-5} \text{ to } 10^{-6}$$

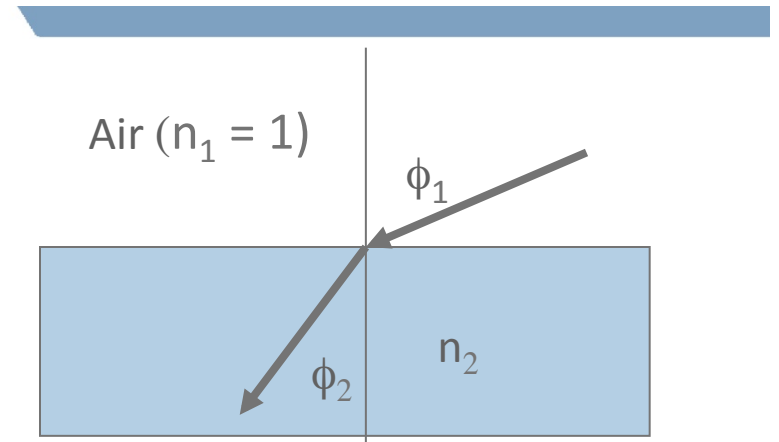
- So you have:
  - an index of refraction less than one
  - differing from unity by only a few ppm

$$V_{\text{group}} = \frac{d\omega}{dk} \quad \text{and} \quad \omega = \frac{ck}{n} \quad \text{so} \quad V_{\text{group}} = \frac{d}{dk} \left( \frac{ck}{n} \right); \quad k = \frac{2\pi}{\lambda} \quad n = 1 - \frac{2\pi n_e r_e}{k^2}$$

$$V_{\text{group}} = \frac{d}{dk} \left[ \frac{ck}{1 - \frac{2\pi n_e r_e}{k^2}} \right] \approx \frac{d}{dk} \left[ ck \left( 1 + \frac{2\pi n_e r_e}{k^2} \right) \right] = c \frac{d}{dk} \left[ \left( k + \frac{2\pi n_e r_e}{k} \right) \right] = c \left( 1 - \frac{2\pi n_e r_e}{k^2} \right)$$

# Snell's Law

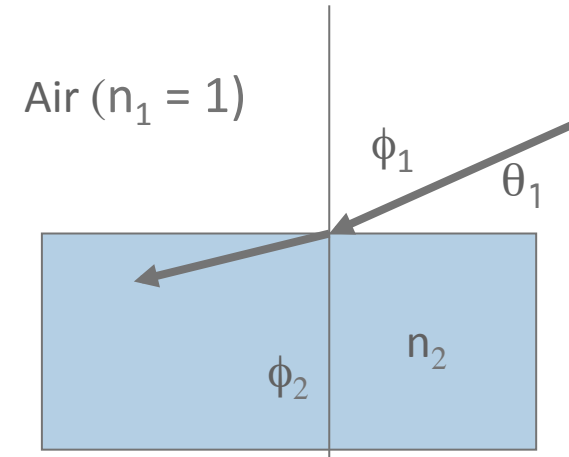
- The reflection and refraction of x-rays can be treated as any other electromagnetic wave traveling in a medium with index of refraction  $n_1$  encountering a boundary with another material with index of refraction  $n_2$ .
- The resultant kinematic properties (which follow from the wave nature of the radiation at boundaries) are:
  - The angle of incidence equals the angle of reflection
  - **$n_1 \sin(\phi_1) = n_2 \sin(\phi_2)$**  (Snell's Law), where the  $\phi$ 's are measured with respect to the boundary normal



Typical values for  $n_2$  (at 5890Å) are:

water:  $n_2 = 1.33$

glass:  $n_2 = 1.52$



For x-rays, the direction of propagation bends away from surface normal.

# Critical Angle for Total External Reflection

- Let an x-ray (in vacuum, where  $n_1 = 1$ ) impinge on a material with index of refraction  $n_2$ . From Snell's Law (when  $\phi_2 = 90^\circ$ ), we have:

$$n_1 \sin(\phi_c) = n_2 \sin(90^\circ) ;$$

$$\cos(\theta_c) = n_2 \cos(0) \quad (\theta = 90^\circ - \phi)$$

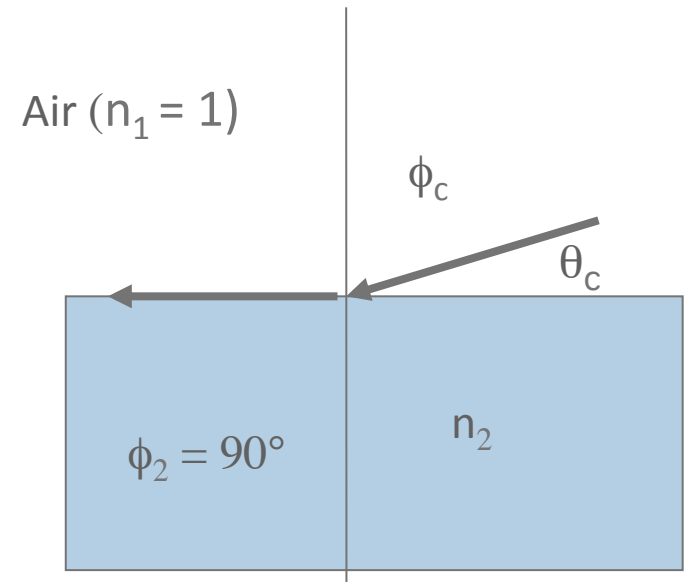
$$\cos(\theta_c) = n_2$$

- Expanding the cosine of a small angle and substituting for  $n_2$  gives:

$$1 - (\theta_c)^2/2 = 1 - \delta$$

$$\theta_c = (2\delta)^{1/2}$$

$\theta_c$  is the so-called critical angle, the angle at which there is total external reflection and the material behaves like a mirror.



Recall that the typical values for  $\delta$  at  $1 \text{ \AA}$  is  $10^{-5}$  to  $10^{-6}$  and so the critical angle is going to be about  $10^{-3}$  or a few milliradians

# X-ray Reflectivity

- The amplitude of the reflected wave can be determined through the Fresnel equations. Sparing you the details, the intensity ratio of the reflected and incident beam is given by:

$$I^R / I = |E^R / E|^2$$

- From the Fresnel equations it can be shown that:
  - Below  $\theta_c$ , there is unit reflectivity (when  $\beta$ , the absorption equals 0)
  - Above  $\theta_c$ , the intensity falls off as  $(\theta_c / \theta)^4$

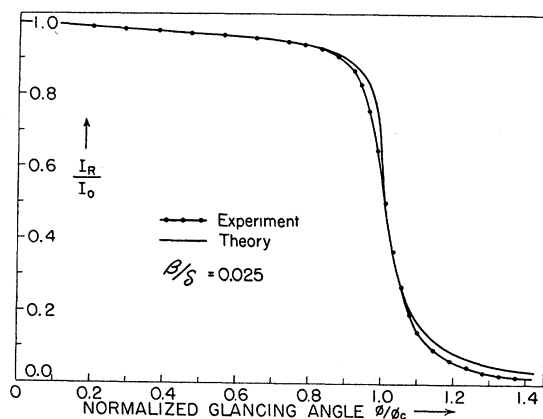
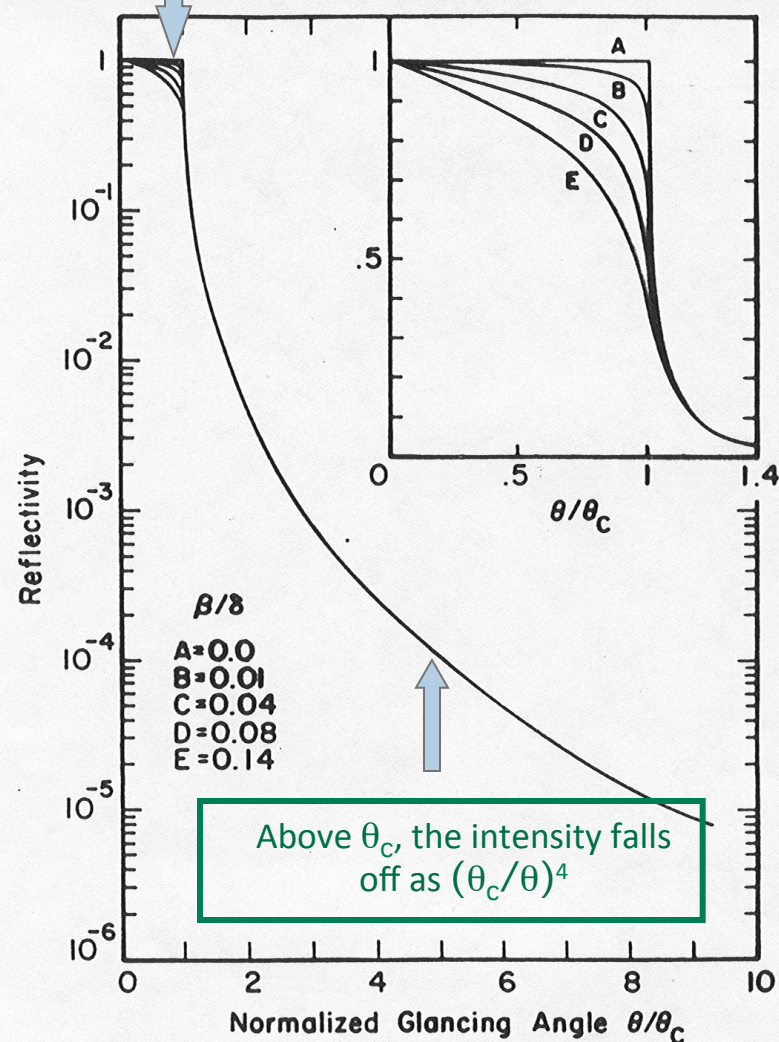


FIG. 2. Intensity reflected from polished glass at small angles of incidence. The angle  $\phi_c$  is the critical angle for total reflection.

Below  $\theta_c$ , there is unit reflectivity (if  $\beta = 0$ )



PHYSICAL REVIEW

VOLUME 95, NUMBER 2

JULY 15, 1954

Surface Studies of Solids by Total Reflection of X-Rays\*

L. G. PARRATT  
Cornell University, Ithaca, New York  
(Received March 22, 1954)



NXSchool 2012

## Energy Cut-off for a Fixed Angle of Incidence

- All the above was developed assuming a single wavelength (monochromatic) incident beam and a variable angle of incidence.
- Often mirrors are used as first optical components. This means a polychromatic incident beam strikes the mirror at some fixed angle.
- The relationship for the critical angle and wavelength can be rewritten in terms of a cut-off energy,  $E_c$ . So for a mirror at a fixed angle  $\theta$ , energies below  $E_c$  will be totally reflected (since the reflectivity is nearly 1) and energies above  $E_c$  will be reduced because of the fall-off of reflectivity:

$$\theta_c = (2\delta)^{1/2}$$

$$\theta = (2(n_e r_e / 2\pi) \lambda^2)^{1/2} = \lambda (n_e r_e / \pi)^{1/2}$$

$$E = hc/\lambda$$

$$E_c = hc/\lambda_c = (hc / \theta) (n_e r_e / \pi)^{1/2}$$

# Uses of Reflective Optics: X-ray Mirrors

- Low-pass filters
  - Since there is an energy cut-off above which radiation is reflected with low efficiency; this means that mirrors can be used to effectively suppress high energies.
  - Depending on the critical energy of the source, mirrors can effectively remove a considerable amount of the heat and reduce the thermal loading on downstream optics.
  - Since in many cases the angle of incidence is fixed (due to the various beamline components downstream), mirrors are designed so that the cut-off energy,  $E_c$  can be varied by having several different coatings deposited on the mirror substrate.

Fix the angle of incidence and vary the material density, i.e.,  $n_e$ .

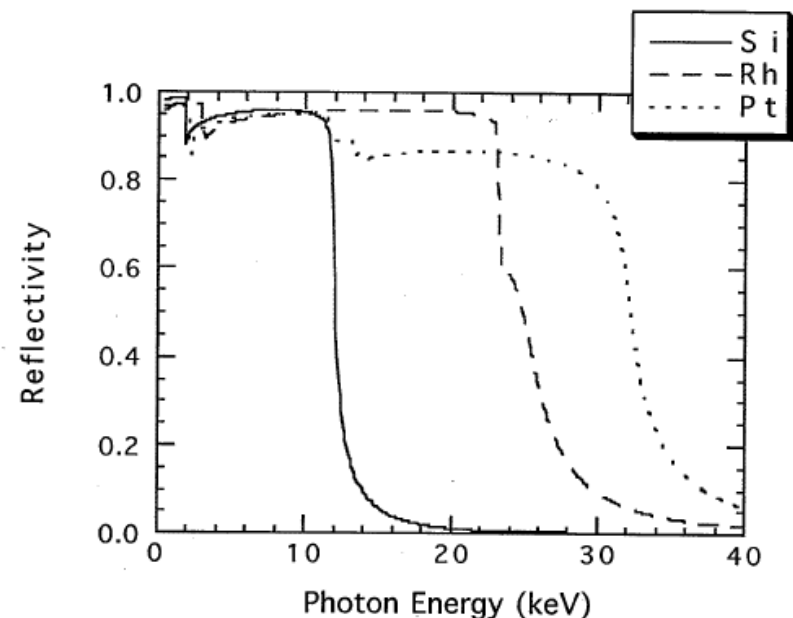
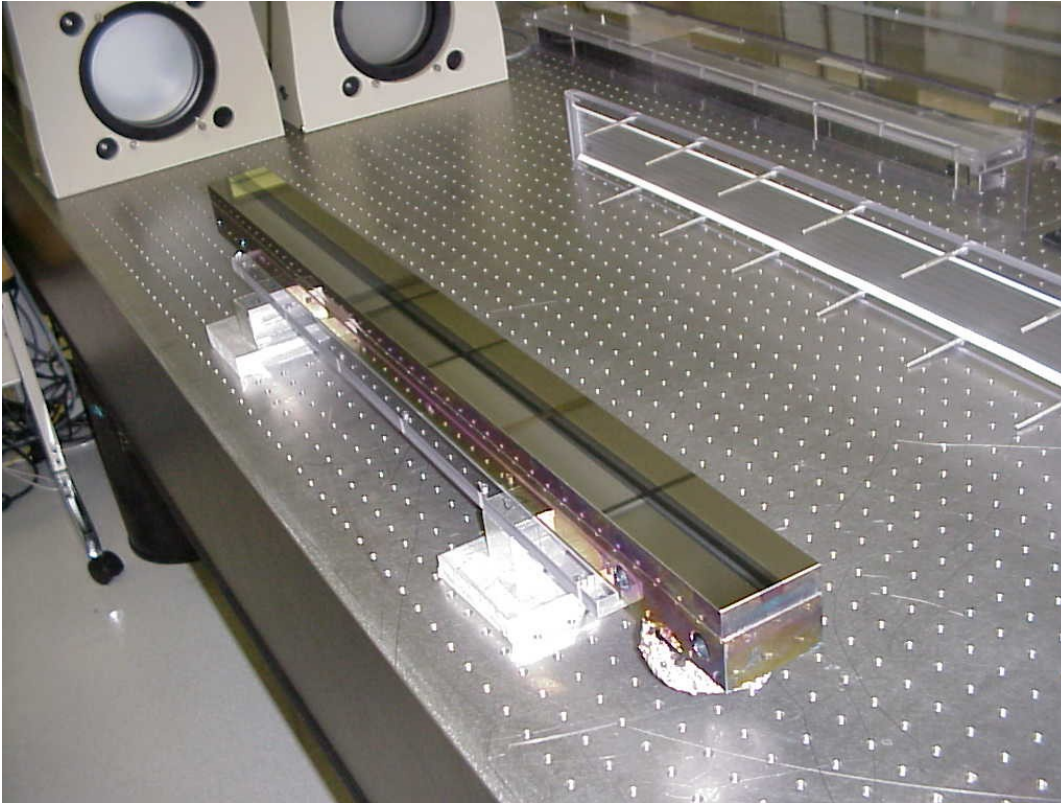


Fig. 2ID-4 X-ray reflectivity as a function of x-ray energy for Si, Rh, and Pt mirrors at  $0.15^\circ$  of incidence angle. Note the cut-off energy for a Pt mirror is about 34 keV.

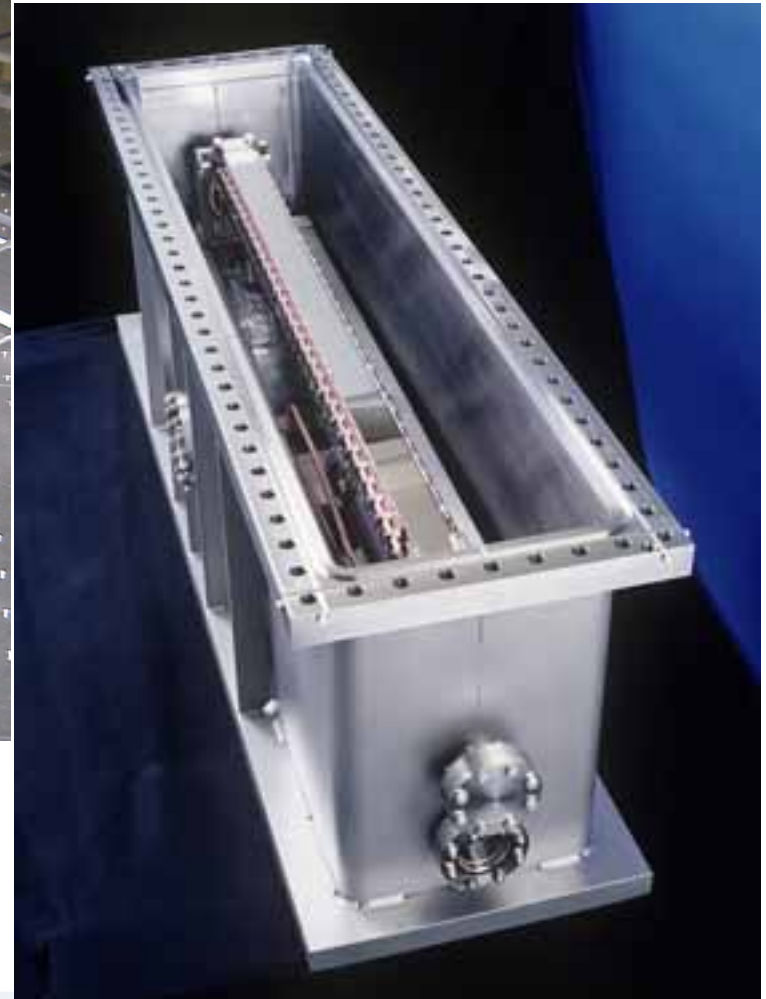


# Consequence of Small Critical Angles- Long Mirrors to Capture Full Beam

Water cooled mirror in  
its vacuum tank.



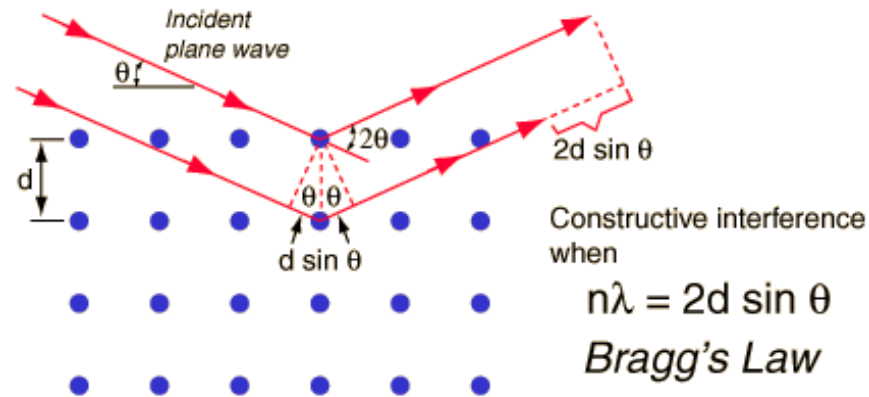
An x-ray mirror that had been in the x-ray beam (you can see the black stripe where the beam hit) being measured for surface roughness off-line.





# Diffractive Optics

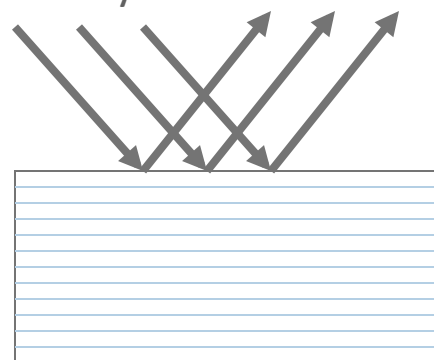
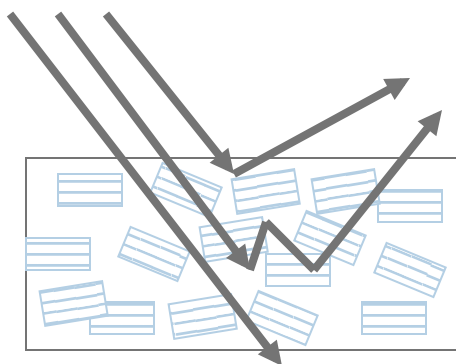
- By far, the most commonly used optical component for x-rays are crystals satisfying Bragg's law, i.e.,



- In nearly all cases, **perfect single crystals** are used as the diffractive elements since:
  - they have a reflectivity near unity (more later)
  - the physics is well understood and components can be fabricated with predicted characteristics
  - they preserve the beam brightness

# Diffraction from Perfect Crystals

- The theory that describes diffraction from perfect crystals is called dynamical diffraction theory (as compared with kinematical theory, which describes diffraction from imperfect or mosaic crystals) first proposed in 1914 by Darwin in two seminal papers.

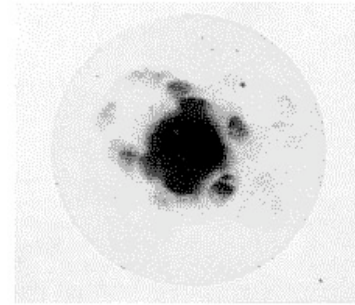


- The name comes from the fact that, during the diffraction process from perfect single crystals, there is a dynamical interplay between the incident and scattered beam, which can be comparable in strength.
- In the case of a strong reflection from a perfect crystal of a monochromatic x-ray beam, the penetration of the x-rays in to the crystal is not limited by the (photoelectric) absorption, but the beam is attenuated due to the reflecting power of the atomic planes. (This type of attenuation is called “extinction”.)

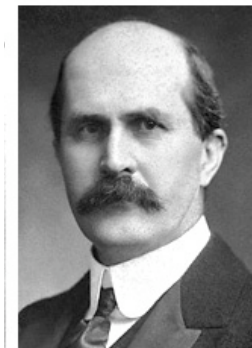
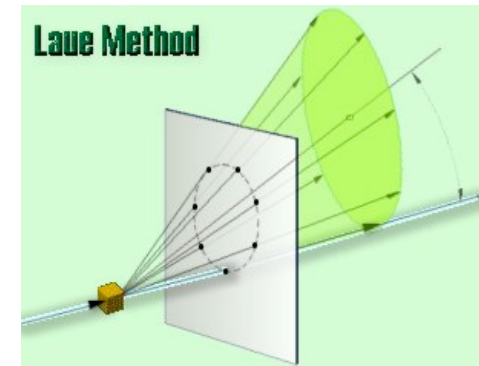
# 2012 - 100 Years of X-ray Diffraction

- Max von Laue (1879-1960) realized that short wavelength electromagnetic rays, such as X-rays, should cause diffraction or interference phenomena in crystals and had worked out the positions of the x-ray interference maxima (reflections) but not the intensity. (M. Laue, Kon. Bay. Ak. 1912, p. 303) based on the work by Friedrich and Knipping (W. Friedrich & P. Knipping, Kon. Bay. Ak. 1912, p. 311).
- Von Laue received Nobel Prize in 1914 for his work.
- William L. Bragg (the son), using his father's spectrometer, reported in 1913 that the scattering phenomena in crystals as the reflection of x-rays from sets of parallel planes of atoms in the crystal. (*"The diffraction of short electromagnetic waves"*, by a crystal. W. L. Bragg. Proc. Camb. Phil. Soc., Vol. XVII, p. 43, 1913).
- The Braggs shared the 1915 Nobel Prize in Physics.

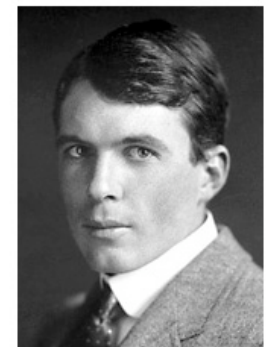
Images from: <http://www4.nau.edu/microanalysis/Microprobe-SEM/History.html>



Max von Laue



William Henry Bragg

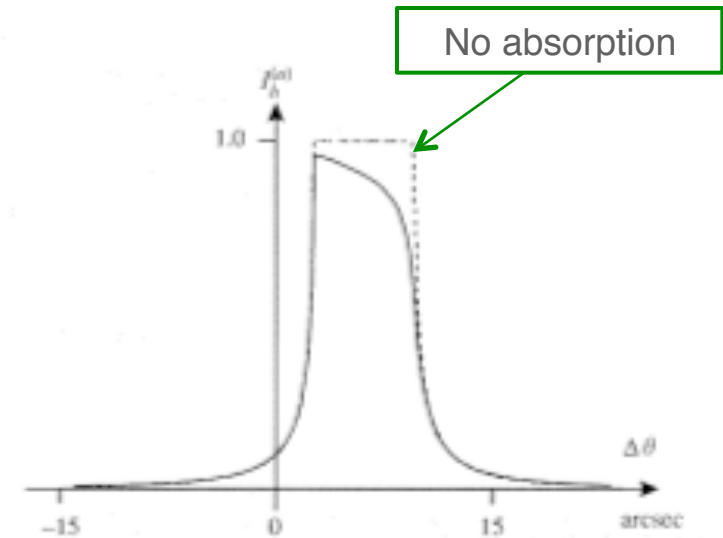


William Lawrence Bragg

# Finite Width of Bragg Reflections- The Darwin Width

- The limited penetration means:
  - the beam only interacts with a limited number of atomic planes
  - the scattered beam gets in and out of the crystal with little loss of amplitude from (photoelectric) absorption.
  
- A consequence of the first item is:
  - There is a finite angular width over which the diffraction occurs. This is often called the **Darwin width**,  $\omega_D$  (after Charles Darwin, the scientist who first worked this out in detail).
  - Depends on the strength of the reflection (hkl) and wavelength.
  
- A consequence of the second item is:
 

The reflectivity over this narrow Darwin width is nearly unity.



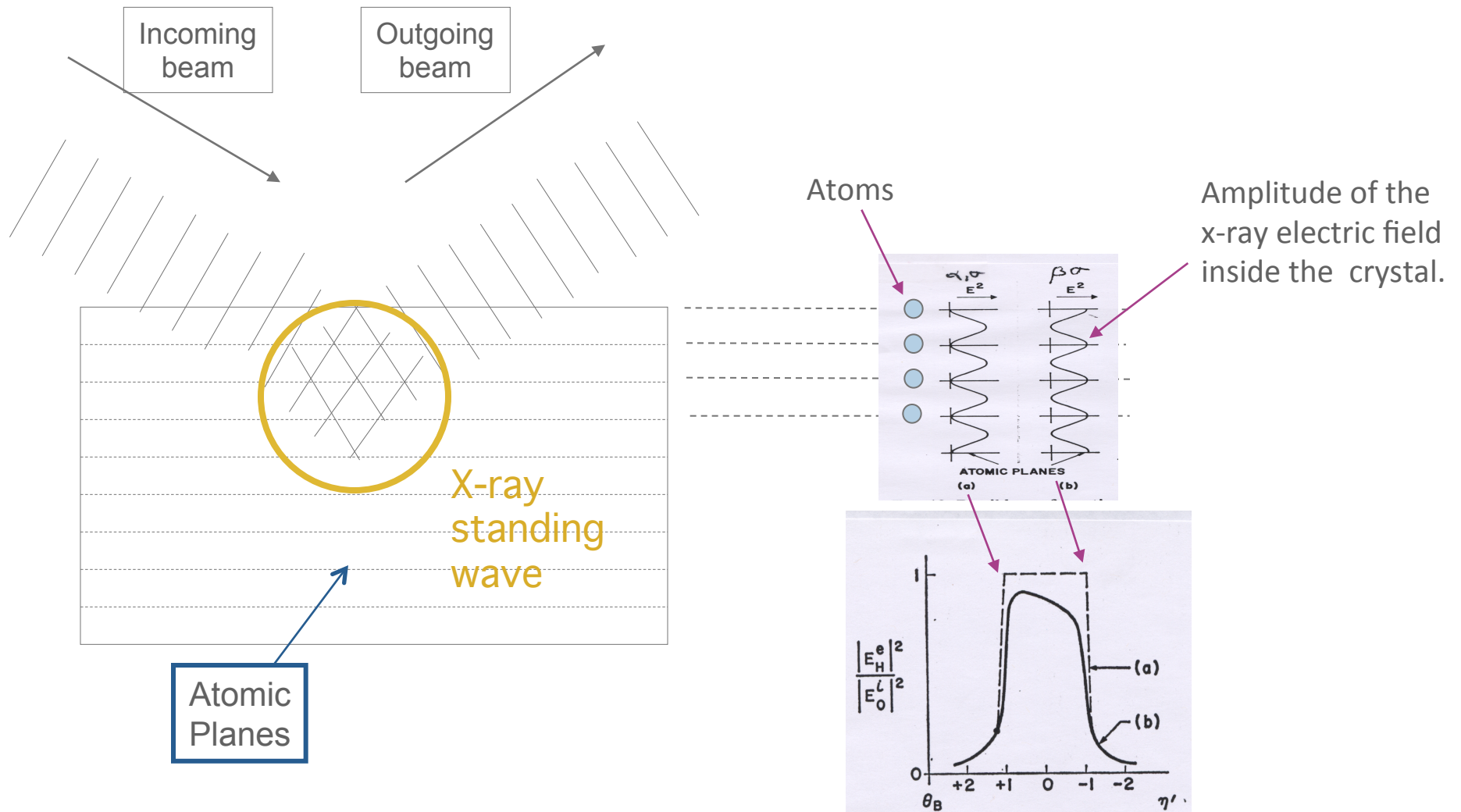
The Darwin width  $\sigma$  polarized x-rays ( $E \perp$  to diffraction plane).

$$\omega_D^\sigma = 2r_e F(hkl) \lambda^2 / \pi V \sin(2\theta)$$

$$\omega_D^\pi = \omega_D^\sigma \cos(2\theta)$$

$F$  = structure factor &  $V$  = volume of unit cell

# X-ray Standing Waves at the Bragg Condition

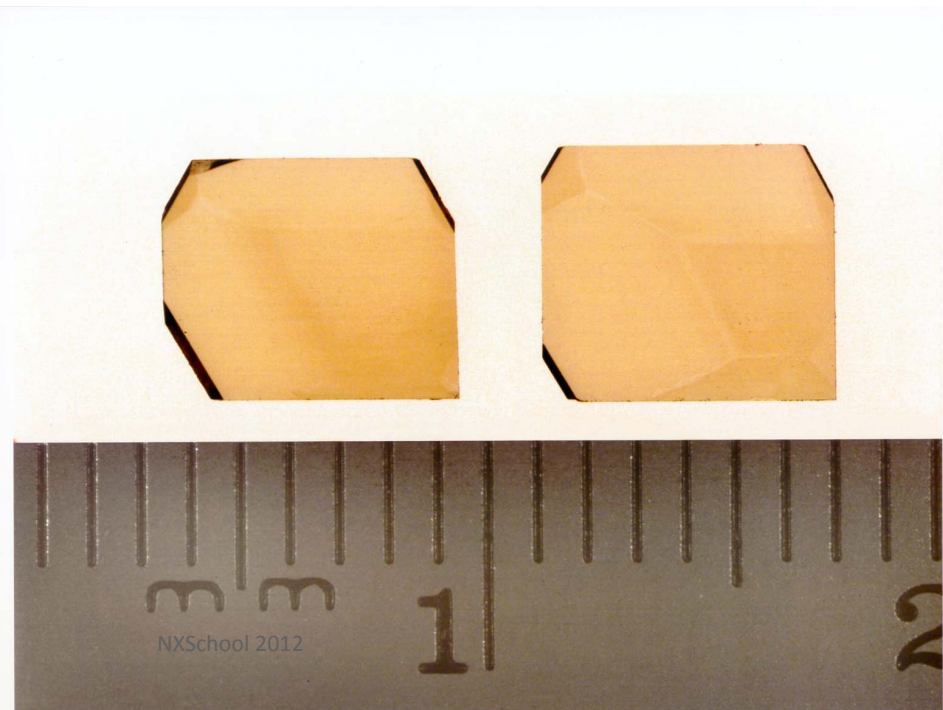


# Real World Perfect Crystals

At first glance, requiring the use of only perfect crystals for x-ray optical components may seem very limiting. However, silicon and germanium, are readily available (due to their use in the semiconductor industry) and are grown in large boules that are relatively inexpensive.



Nearly perfect single crystals of synthetic (grown) diamonds are also desirable, primarily for their mechanical properties which are extremely important when used as first optical components.





# Perfect Crystal Monochromators

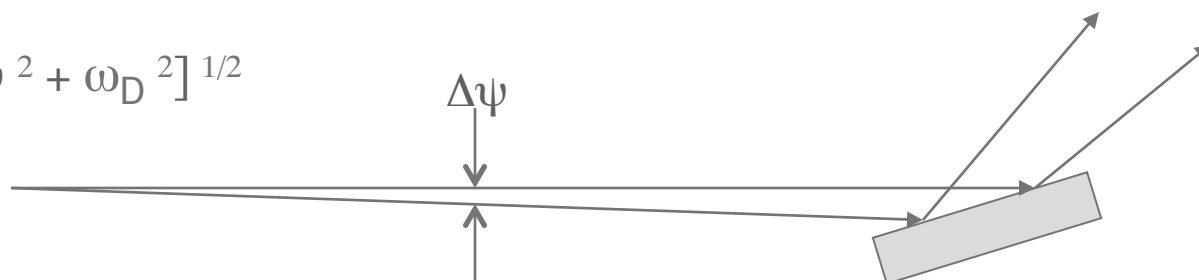
- The most frequent use of perfect crystal optics are for **x-ray monochromators**. They simply use Bragg's Law to select a particular wavelength (or energy, since  $\lambda = hc/E$ ):

$$\lambda = 2d \sin(\theta).$$

- If we differentiate Bragg's Law, we can determine the energy resolution of the monochromator.

$$\Delta\lambda / \lambda = \Delta E / E = \cot(\theta) \Delta\theta$$

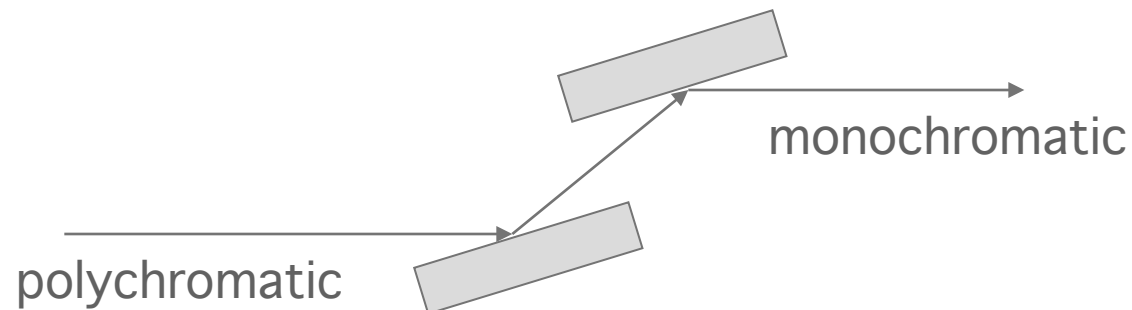
$$\Delta\theta = [\Delta\psi^2 + \omega_D^2]^{1/2}$$



- A value of  $\omega_D$  for the (111) reflection in silicon at 8 keV ( $1.5\text{\AA}$ ) is about 8 sec of arc or 40 microradians. Recall that, for an undulator, the opening angle is about 10 - 15 microradians. Here the energy resolution of the mono is determined by the crystal.

# Double Crystal Monochromators

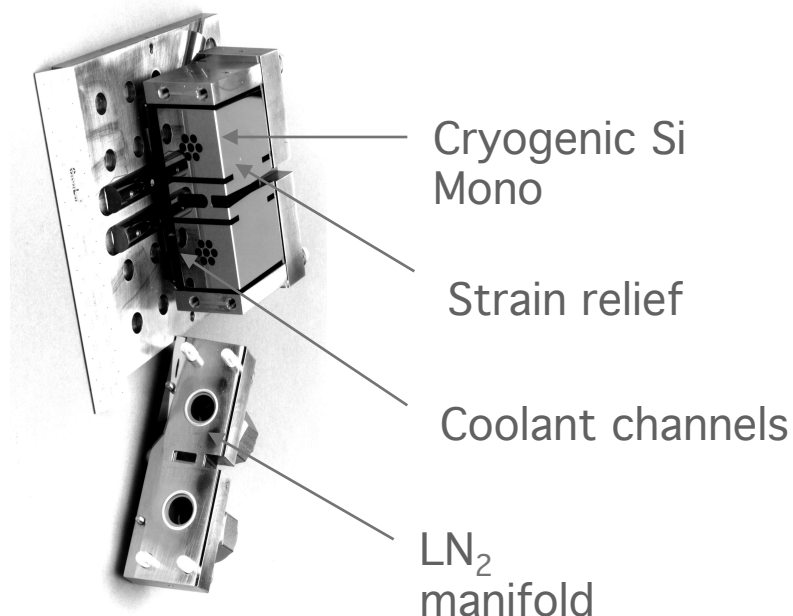
- The most common arrangement for a monochromator is the **double-crystal monochromator**. It:
  - is non-dispersive, that is all rays that diffract from the first crystal simultaneously diffract from the second crystal (if same crystals with same hkl's are used)
  - keeps the beam fixed in space as the energy is changed.
- There is little loss in the throughput because the reflectivity is near unity over the Darwin width.





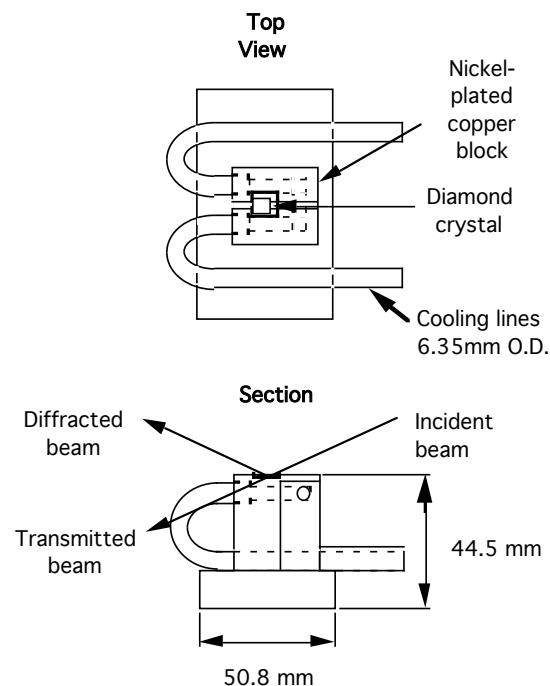
## Cooled Monochromators

- One solution to this problem is to cool the Si crystals to liquid nitrogen temperatures for that increases the thermal conductivity and reduces the coefficient of thermal expansion.



Cryogenically Cooled Si Mono

## Water Cooled Diamond Mono

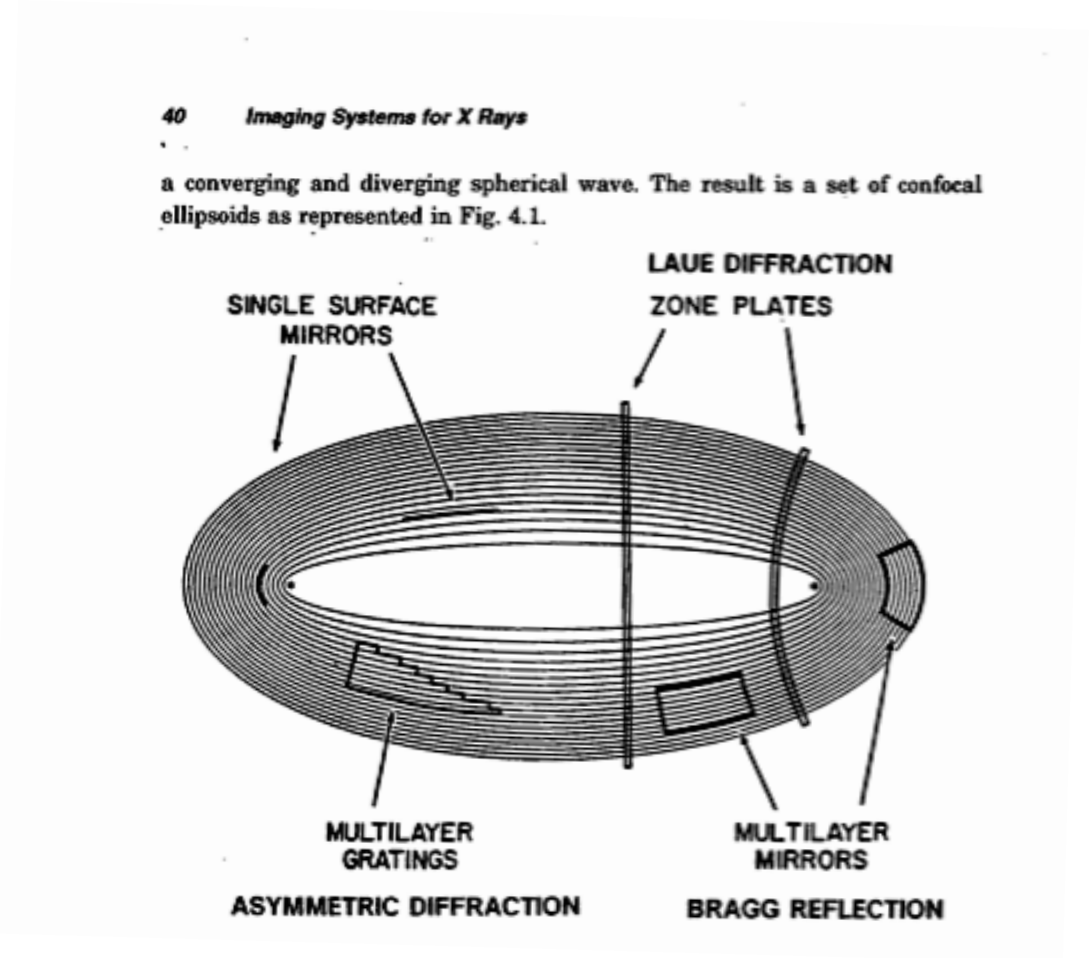


Another solution to this thermal problem is to go to other materials (i.e., diamond) that have better thermal and mechanical properties than Si at room temperature.

See Appendix 2 for information regarding thermal issues for monochromators.

# Focusing X-ray Optics

- There is considerable interest in focused x-ray beams from 100's of microns to ten's of nanometers in size.
- Focused beams can be used in a variety of techniques:
  - Small samples
  - Extreme conditions
  - Scanning microscopies
- There are a variety of focusing optical components:
  - curved crystals (diffractive)
  - curved mirrors (reflective)
  - zone plates (diffractive)
  - multilayer Laue lenses (diffractive)
  - compound refractive lenses (refractive)



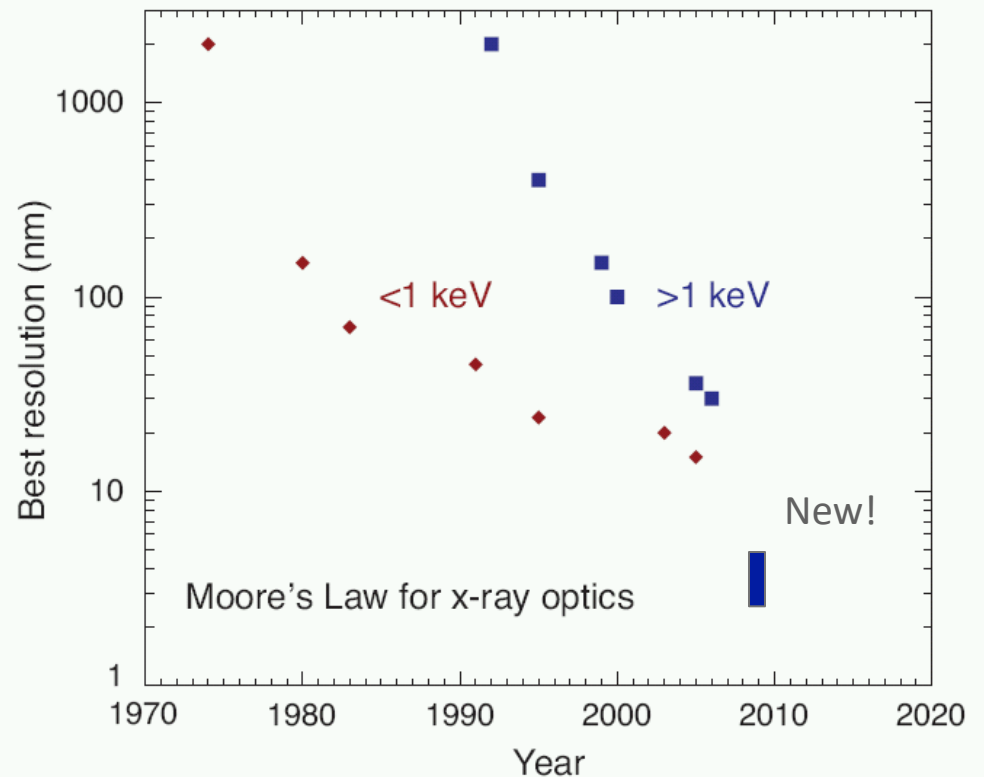
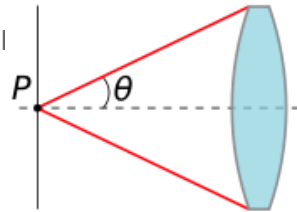
# State of the Art Focusing of X-rays

- Since X-rays are electromagnetic waves, the spatial resolution limit should be:

$$\Delta r = 0.6 \lambda / NA$$

where the numerical aperture

$$NA = n \sin(\theta)$$

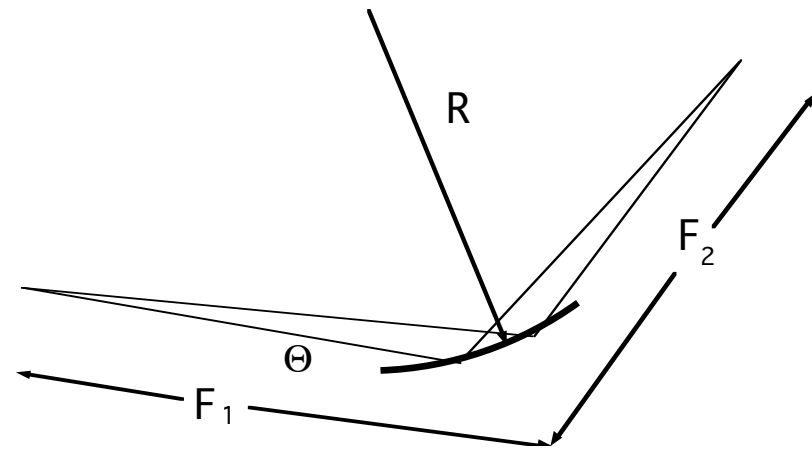


- Current State of the art:

Refractive Optics:	CRLs	$d \sim 50 \text{ nm}$ ( $E = 21 \text{ keV}$ ) ( <i>Schroer, APL, 2005</i> )
Reflective Optics:	Mirror (1D)	$d \sim 7 \text{ nm}$ ( $E = 20 \text{ keV}$ ) ( <i>Mimura et al, Nature Physics, 2009</i> )
Diffraction Optics:	Zone Plates	$d \sim 17 \text{ nm}$ , ( $E = 10 \text{ keV}$ ) ( <i>Comamala et al. JSR, 2012</i> )
	MLLs (1D)	$d \sim 16 \text{ nm}$ , ( $E = 20 \text{ keV}$ ) ( <i>Kang et al, APL, 2008</i> )

# Reflecting Focusing Optics: Mirrors

- One-dimensional focusing, collimating, etc.
  - An ellipse is the ideal shape for a reflecting surface for point-to-point focusing. (A source at one foci will be imaged at the other foci.)
  - Collimation can be achieved by a parabola if the source is placed at the focal point. (This is simply an ellipse with the second focal point at infinity.)
  - In many cases cylindrically shaped mirrors are used rather than ellipses and parabolas since they are considerably easier to fabricate.



Focal length,  $f = [R_m \sin \theta]/2$

The “lensmaker” equation is:

$$\frac{1}{f} = \frac{1}{F_1} + \frac{1}{F_2}$$

So:

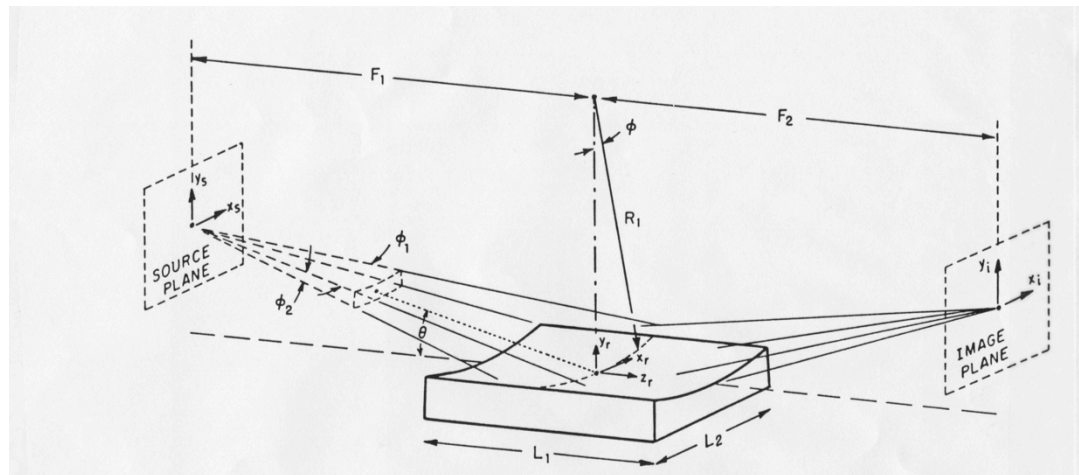
$$R_m = [2/\sin \theta] [F_1 F_2 / (F_1 + F_2)]$$

Typically,  $\theta = 3 \text{ mrad}$ ,  $F_1 = 30\text{m}$ ,  
 $F_2 = 30\text{m}$  and so  
 $R_m = 10 \text{ kilometers}$

# Focusing in Two Dimensions with Mirrors

- Two-dimensional focusing (toroids and ellipsoids)
  - An ellipsoid is the ideal shape for a reflecting surface for point-to-point focusing.
  - Bent cylinders are often used in place of an ellipsoid.
  - The sagittal radius,  $R_s$ , is given by:

$$R_s = R_m \sin^2 \theta$$

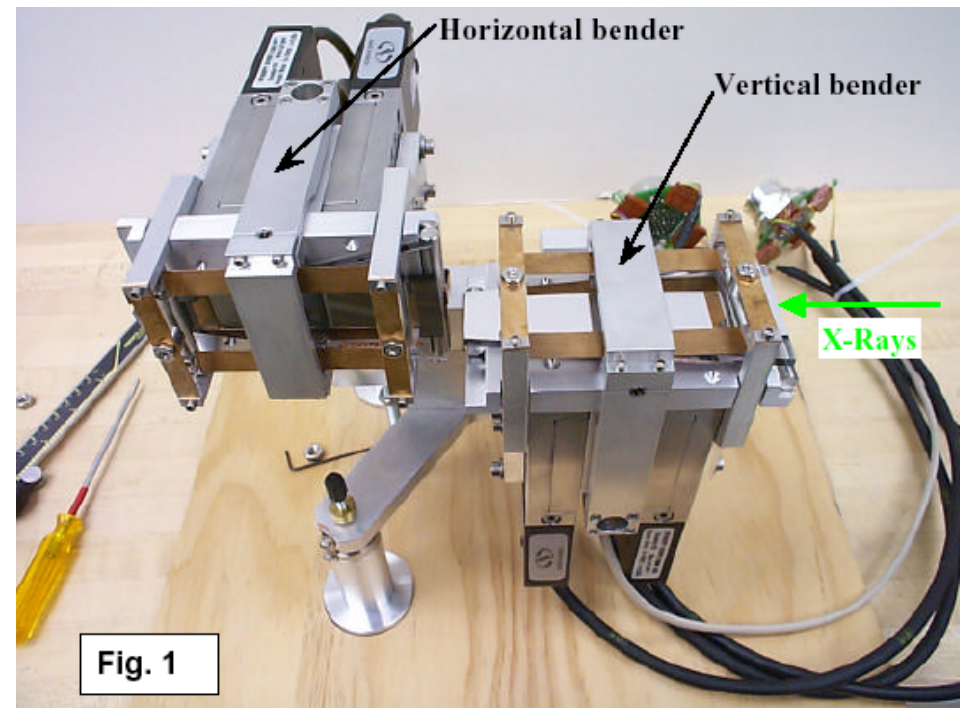
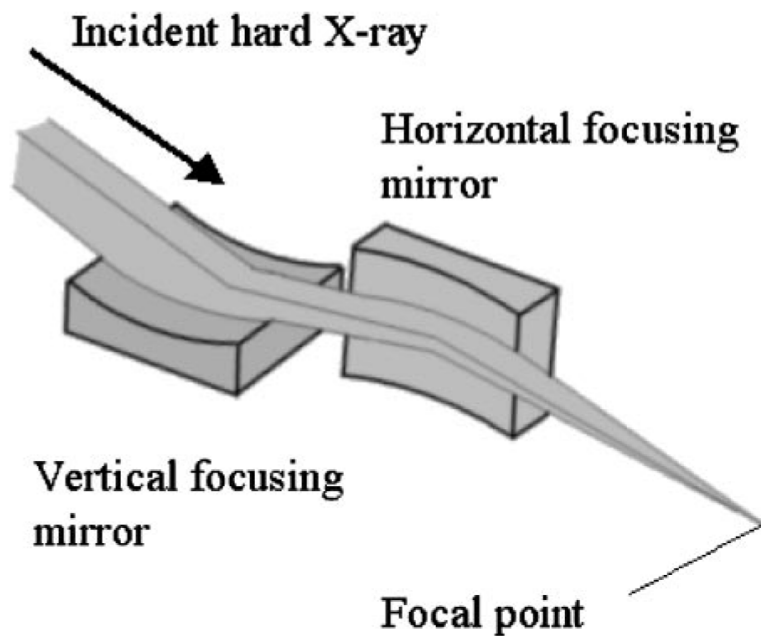


- In our example, from the last slide,  $q = 3$  mrad and  $R_m = 10$  km so the sagittal radius would be:

$$R_s = 9 \text{ cm}$$

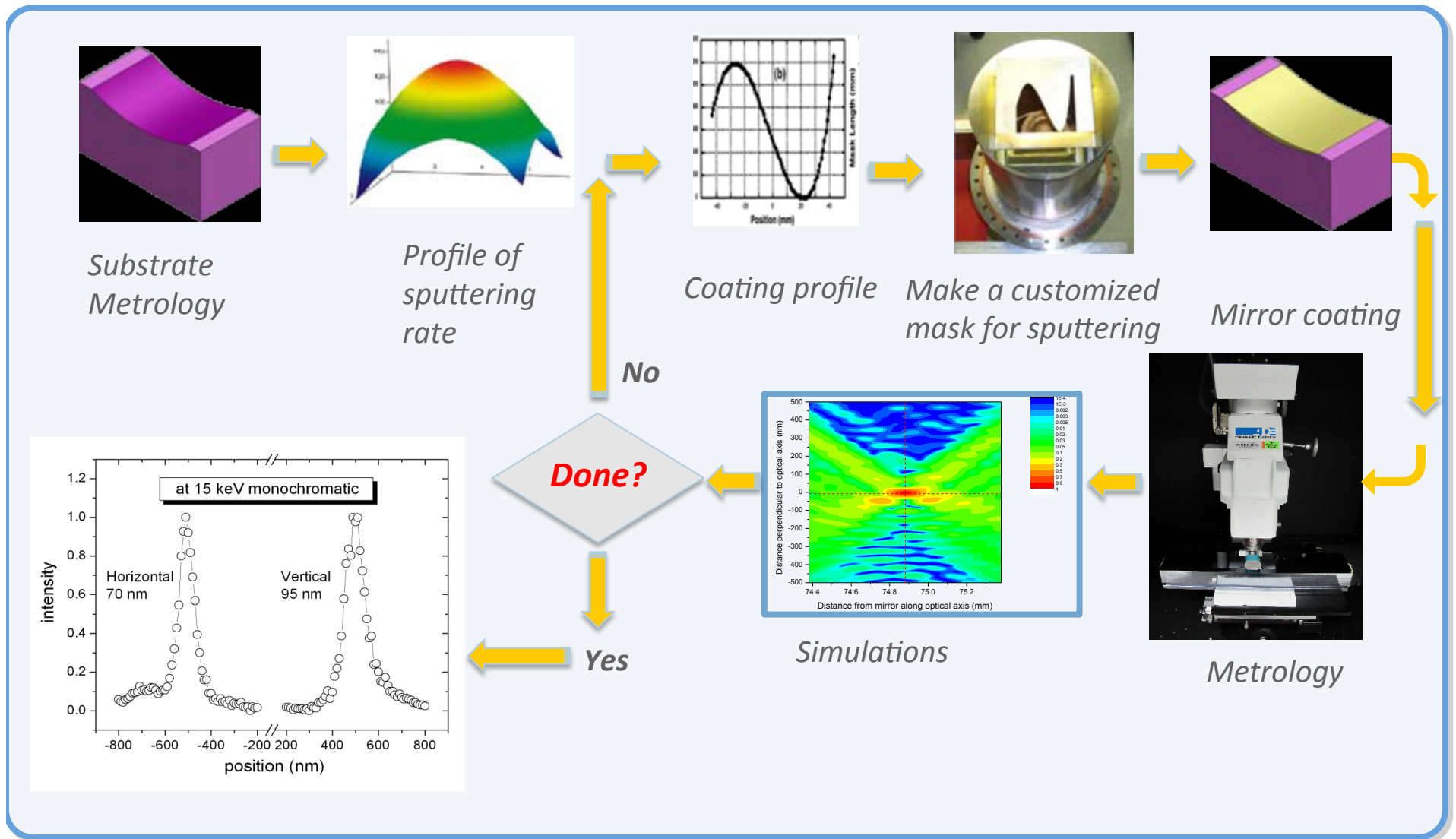
# Focusing in Two Dimensions - KB Systems

- Another system which focuses in two dimensions consists of a set of two orthogonal singly focusing mirrors, off which incident X-rays reflect successively, as first proposed in 1948 by Kirkpatrick and Baez.
- This system allows for easier fabrication of the mirrors and is used frequently at synchrotron sources.



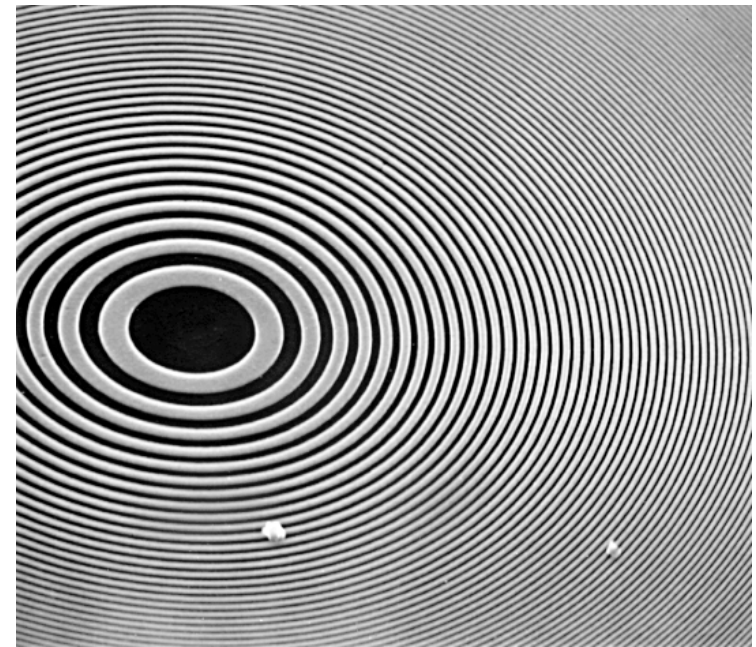


# Fabrication of KB Mirrors by Profile-coating



# Diffractive Focusing Optics: X-ray Zone Plates

- Zone plates are diffraction gratings, that is, structures composed of alternating concentric zones of two materials with different (complex) refractive indices.
- The focusing capability is based on constructive interference of the wavefront modified by passage through the zone plate.
- The wave that emerges from the zone plate is the superposition of spherical waves, one from each of the zones.
- The wavefront modification is obtained through the introduction of a relative change in amplitude or phase in the beams emerging from two neighboring zones.



JOURNAL OF THE OPTICAL SOCIETY OF AMERICA

VOLUME 51, NUMBER 4

APRIL, 1961

## Fresnel Zone Plate for Optical Image Formation Using Extreme Ultraviolet and Soft X Radiation

ALBERT V. BAEZ

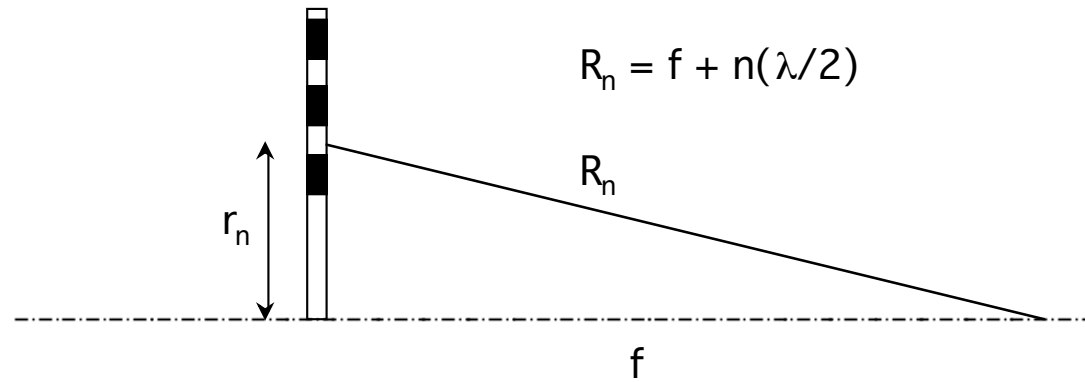
*Smithsonian Astrophysical Observatory, Cambridge, Massachusetts*

(Received April 25, 1960)



# Zone Plate Physics

Condition so the pathlength varies by  $\lambda/2$  for each ring.



$$R_n = f + n(\lambda/2)$$

The radius of the  $n$ th zone is therefore:

$$r_n = (R_n^2 - f^2)^{1/2} = [(f + n(\lambda/2))^2 - f^2]^{1/2}$$

$$= [nf\lambda + n^2(\lambda^2/4)]^{1/2}$$

If  $f \gg n\lambda$ , as is usually the case with hard x-rays, then:

$$r_n = (nf\lambda)^{1/2}$$

# Focal Length and Focus Spot Size

- The focal length for the  $m^{\text{th}}$  order can be written as:

$$f_m = r_n^2 / m\lambda n$$

- In general, the size of the focal spot from the zone plate is determined by the width of the outermost ring,  $\Delta r_{\text{out}}$ , and is given by:

$$\Delta x = 1.22 \Delta r_{\text{out}} / m .$$

- Zones plates with outermost ring widths of less than 30 nanometers can currently be fabricated for soft x-rays.

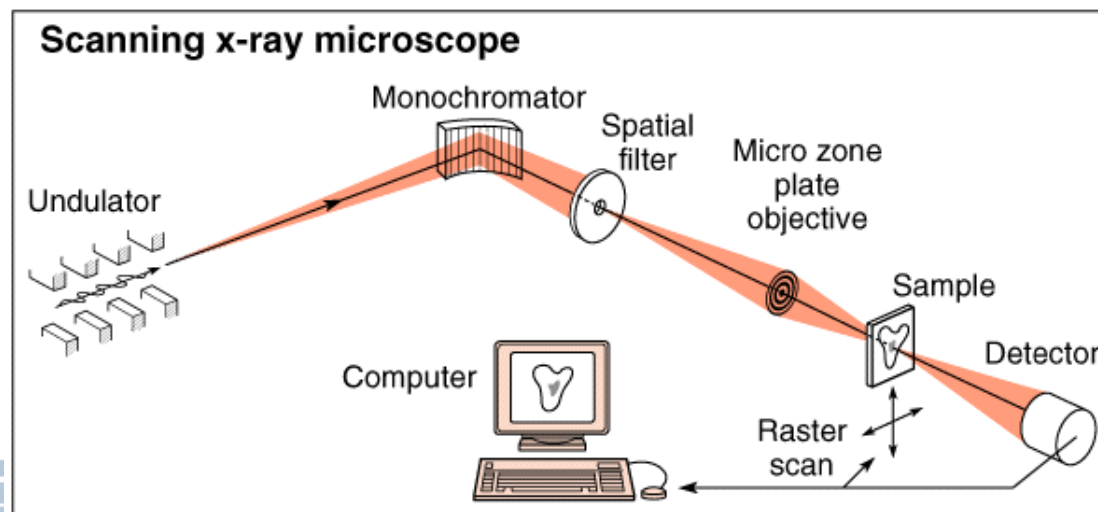


Figure from Center  
for X-ray Optics  
LBNL

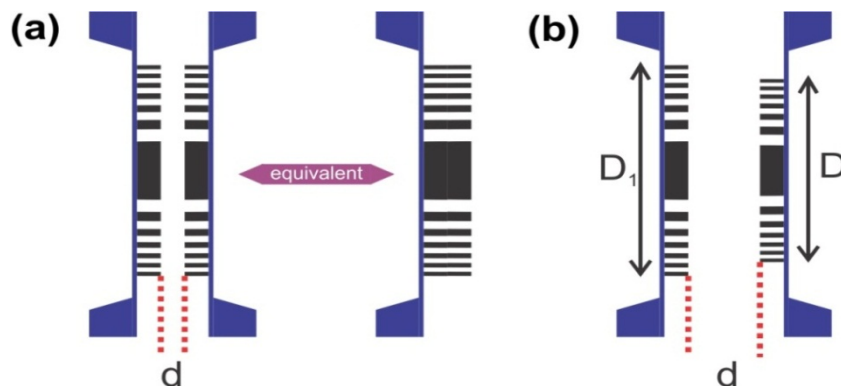
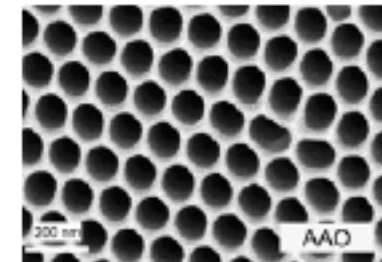
# Hard X-ray Phase Zone Plates

- The difficulty with making zone plates at hard x-ray energies is one of fabrication. You need:
  - small width outermost zone for focusing (less than 100 nms)
  - but it has to be thick (high) to totally absorb the unwanted waves
  - i.e. the aspect ratio (height/width) is very large -  $10^2$  to  $10^3$  and therefore difficult to fabricate
- An alternative to “blocking” out those rays that are out of phase (as in an amplitude zone plate), the thickness of the material can be adjusted so that the wave experiences a phase shift of  $\pi$ .
- Phase zone plates have a much better efficiency than amplitude zone plates (10% efficiency for amplitude zone plates vs 40% for phase zone plates).
- The phase zone plates ease the thickness requirement (as compared to the amplitude zone plates) but the aspect ratio is still an issue.

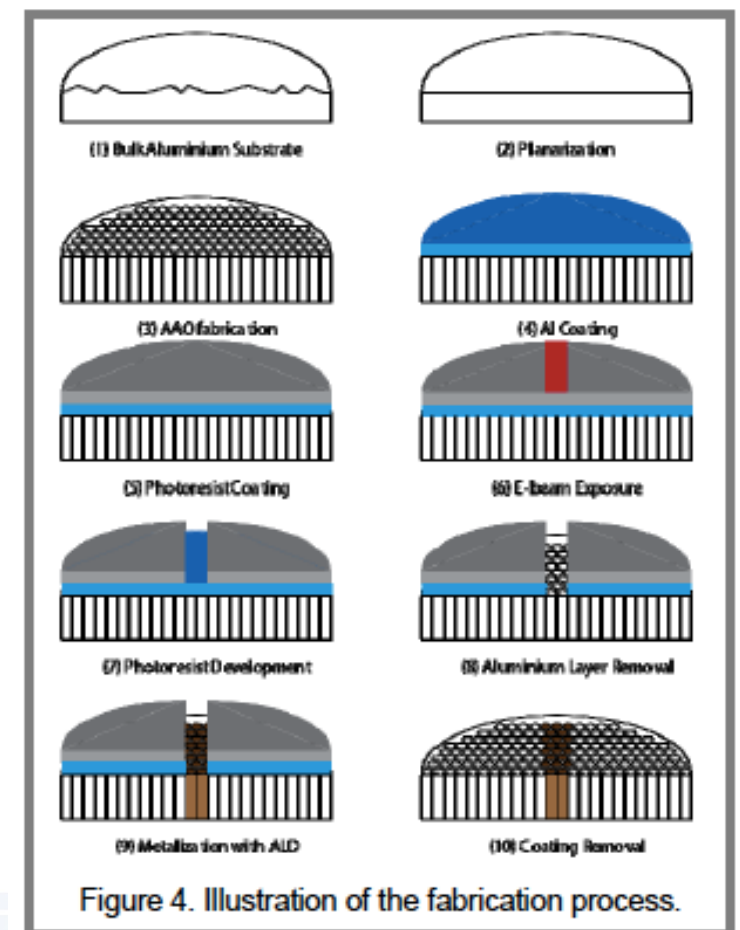


# Zone Plate Optics Development at the APS

- APS has a research program to develop hard x-ray zone plates for 10-100 keV using:
  - e-beam Lithography on Anodic Aluminum Oxide and Atomic Layer Deposition (AAO ALD) and
  - zone doubling via Center for Nanoscale Materials

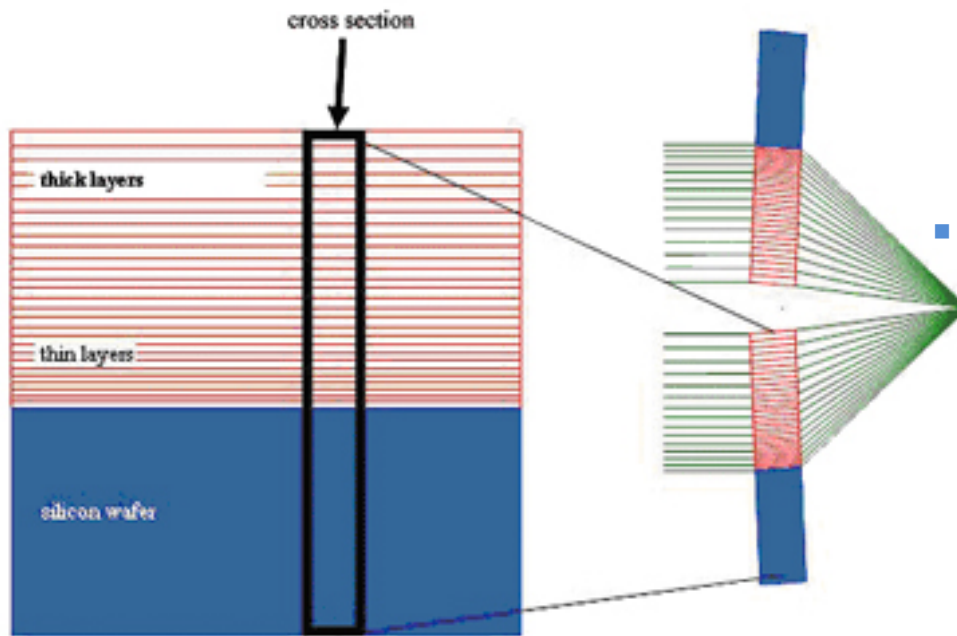


Identical FZPs can be stacked to obtain an equivalent thicker structure with a substantially increased the diffraction efficiency

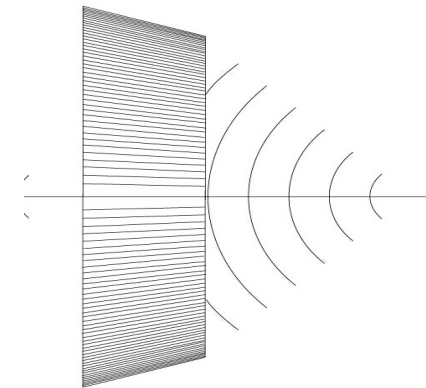


# A New Approach to Fabricating Zone Plates

- Start with a linear zone plate geometry and then use a Kirkpatrick-Baez configuration to get focusing in both directions.
- Using state-of-the-art deposition techniques, start with the thinnest layer first and fabricate a multilayer structure with the layer spacing following the Fresnel zone plate rule.
- Slice and polish the multilayer structure to get a linear zone plate.



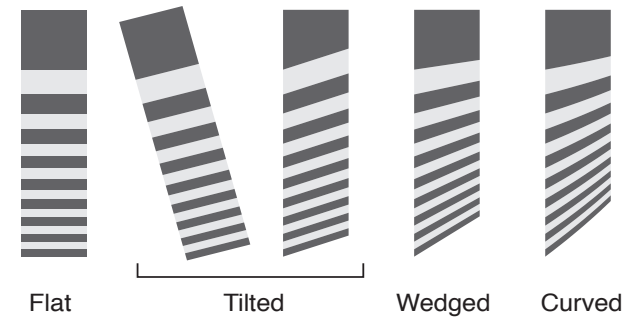
- Each MLL comprises 1,588 layers (lines)
- The thinnest layer (line) is 5 nanometers thick
- The MLL has a current focus of 16 nanometers @ 19 keV!



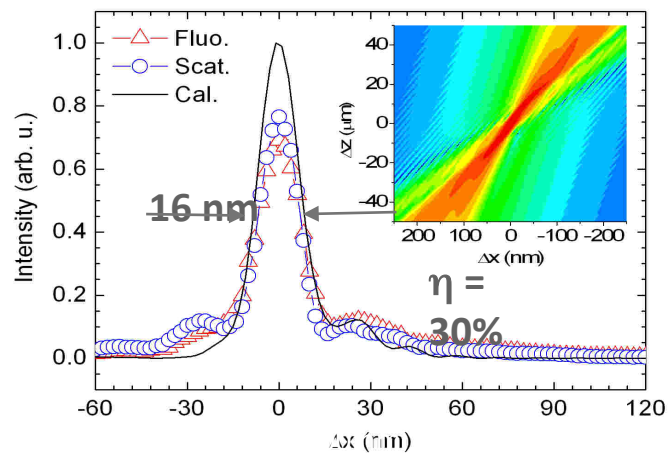
Wedged MLL

# Multilayer Laue Lenses

- Technical approach
  - Crossed multilayer-based linear zone plate structure

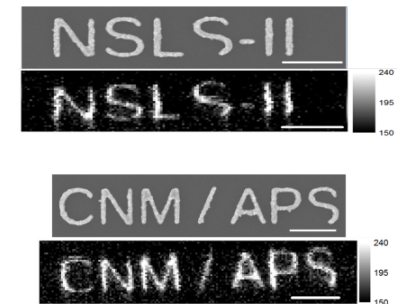
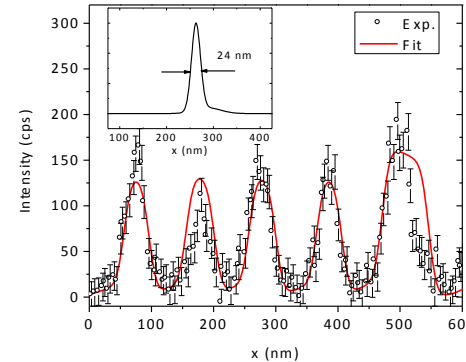


## 1-D focus: 16 nm (half MLL) @ 19.5 keV



Kang et al, APL 92, 221114 (2008)

## 2-D focus: 25 x 40 nm<sup>2</sup> (2D, crossed MLL) @ 20 keV

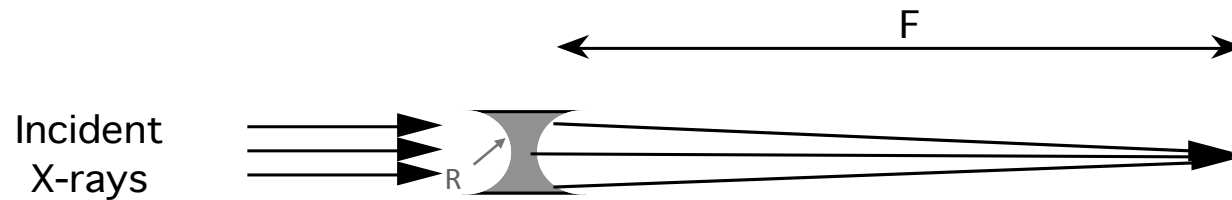


2-D imaging with crossed MLL (ANL/BNL collaboration)

Yan et al, H. Yan et al, Opt. Exp. 2011

# Refractive Focusing Optics: X-ray Lenses

- Roentgen's first experiments convinced him that x-rays could not be concentrated by lenses; thirty years later his successors understood why.
- Refractive lenses were considered by Kirkpatrick and Baez in 1948 for focusing but were abandoned for crossed mirrors.



- What has happened over the last several years that now makes the compound lens not on feasible but now routinely used? The answer is high brightness synchrotron radiation beams.

# Compound Refractive Lenses

The Lens Maker's Equation:

$$1/F = \delta (1/R_1 + 1/R_2 + \text{etc.})$$

For a single lens:

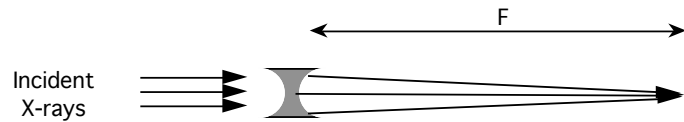
$$1/F = \delta(1/R + 1/R)$$

or

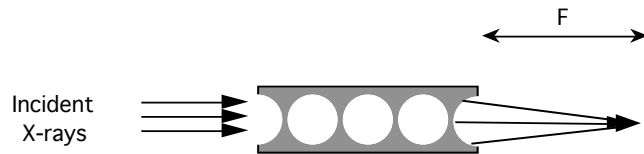
$$F = R / 2\delta$$

If we have N surfaces, all with radius r:

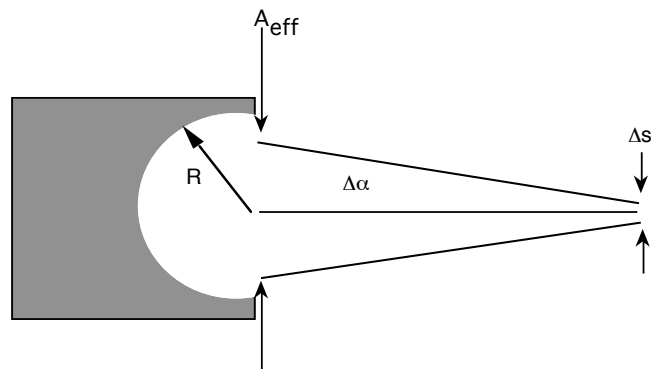
$$F = R/2N\delta$$



Single Refractive Lens



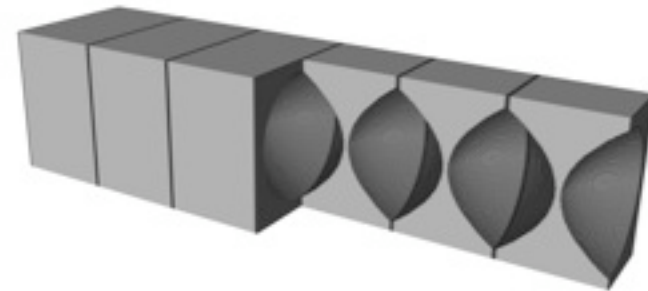
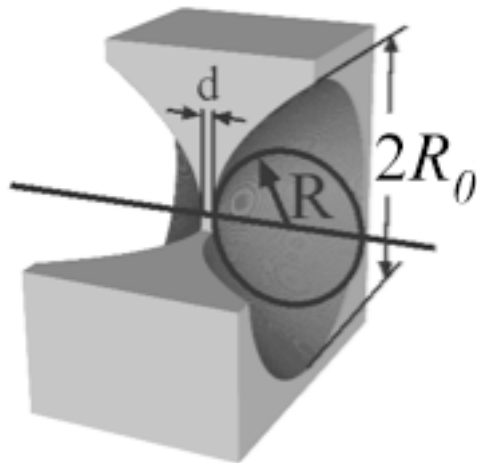
Compound Refractive Lens



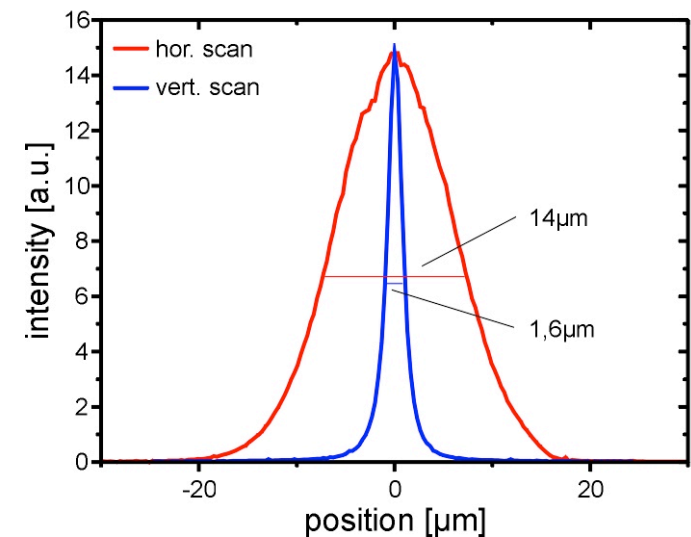
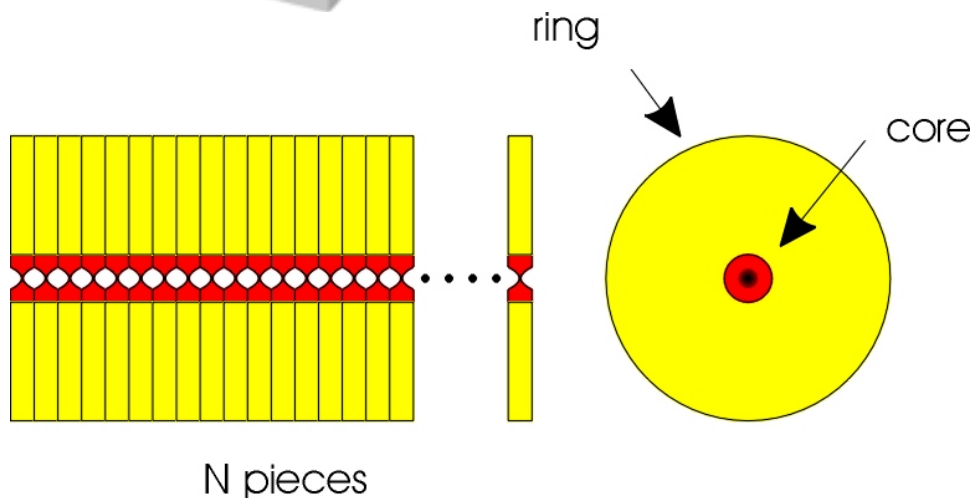


# Focusing in Two Dimensions with Refractive Lenses

- Using spheres but increases the difficulty of fabrication. Paraboloids are the shapes we really want to eliminate spherical aberrations.



[tu-dresden.de/.../isp/skm/research/xray\\_lenses](http://tu-dresden.de/.../isp/skm/research/xray_lenses)



# Why Are Refractive Lenses Effective at Synchrotron Sources?

- Unfortunately materials of large  $d$  are also strong absorbers, the absorption coefficient increasing much more rapidly than  $\delta$  with increasing atomic number. An element of low atomic number, such as beryllium, is indicated.
- Plugging in some numbers, suppose that:
  - $R = 1 \text{ mm}$
  - $\delta \approx 10^{-6}$
  - $N = 50$
- Then the focal length,  $F$ , would be at 10 m.
- These lenses focus at rather larger distances and are well adapted to the scale of synchrotron radiation beamlines.

# Comparison of Focusing Optics

Type of Optic	FZP	MLL	K-B Optics	Refractive Lenses
Typical energy (keV)	5 to 25	> 15	5 to 25	Be 6 - 40 keV Al 40 - 80 keV Ni 80 - 150 keV
Present spot size	25 nm*	16 nm (1D)* 25 x 40 nm <sup>2</sup> (2D)*	30 x 50 nm <sup>2**</sup> 70 x 95 nm <sup>2*</sup>	< 100 nm
Fabrication limit	~10 nm	~2 nm	~25 nm	??
Demonstrated efficiency	6% @ 10 keV	30% (1D) 17% (2D)	Unspecified	1% to 40%
Acceptance (normal-incidence-equivalent )	~250 μm diam	~40 μm	~350 μm**	~100-350 μm

\*Achieved at APS (MLL in collaboration with NSLS-II; KB Mirrors are of Montel type)

\*\*Achieved by the Osaka/Spring-8 team (Not achievable with the current APS beamlines)

# Summary

- X-ray optics is still an active area of research at both universities and national laboratories.
- **New nano-fabrication capabilities** are opening up new possibilities for focusing components.
- Also availability of **high quality materials** (such as dislocation-free diamond in large sizes).
- Metrology is key to making good optics – “**You Can’t Improve What You Can’t Measure**”

LETTERS

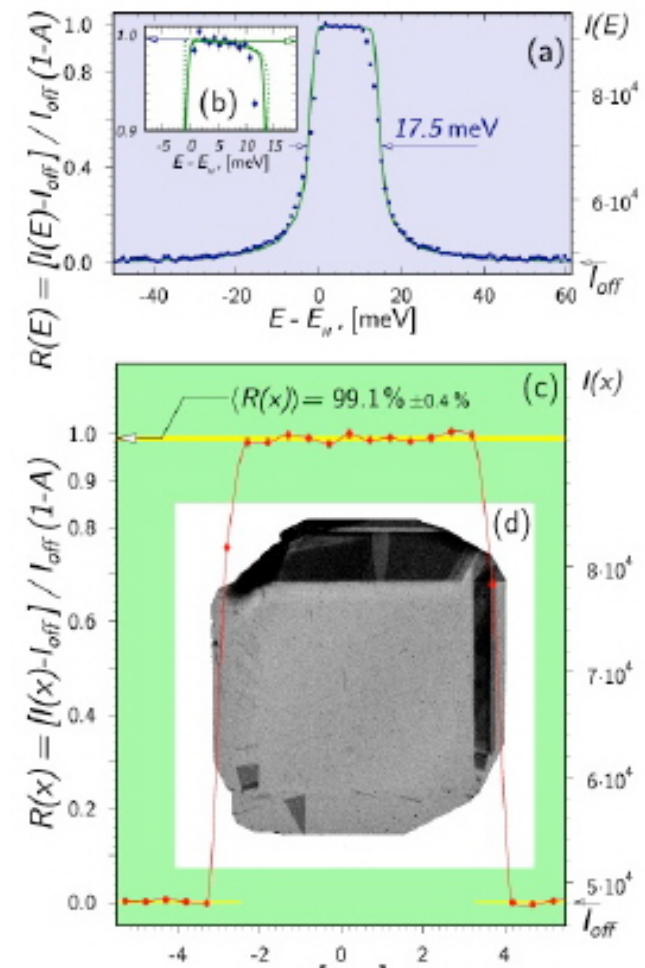
PUBLISHED ONLINE: 22 NOVEMBER 2009 | DOI:10.1038/NPHYS1457

nature  
physics

## Breaking the 10 nm barrier in hard-X-ray focusing

Hidekazu Mimura<sup>1\*</sup>, Soichiro Handa<sup>1</sup>, Takashi Kimura<sup>1</sup>, Hirokatsu Yumoto<sup>2</sup>, Daisuke Yamakawa<sup>1</sup>, Hikaru Yokoyama<sup>1</sup>, Satoshi Matsuyama<sup>1</sup>, Kouji Inagaki<sup>1</sup>, Kazuya Yamamura<sup>3</sup>, Yasuhisa Sano<sup>1</sup>, Kenji Tamasaku<sup>4</sup>, Yoshinori Nishino<sup>4</sup>, Makina Yabashi<sup>4</sup>, Tetsuya Ishikawa<sup>4</sup> and Kazuto Yamauchi<sup>1,3</sup>

NXSchool 2012



LETTERS

PUBLISHED ONLINE: 17 JANUARY 2010 | DOI:10.1038/NPHYS1506

nature  
physics

## High-reflectivity high-resolution X-ray crystal optics with diamonds

Yuri V. Shvyd'ko<sup>1\*</sup>, Stanislav Stoupin<sup>1</sup>, Alessandro Cunsolo<sup>1,2</sup>, Ayman H. Said<sup>1</sup> and Xianrong Huang<sup>2</sup>

# Appendix 1: Dielectric Constant

The dielectric constant,  $\kappa$ , is defined as follows:

$$\kappa = D/E = (E + 4\pi P)/E = 1 + 4\pi(P/E)$$

For a single electron:

$$p = -ex$$

and for multiple electrons:

$$P = -exn_e$$

where  $n_e$  is the number of electrons/unit volume. In a simple harmonic approx:

$F = ma = m\ddot{x} = -eE - kx$  where  $k$  is the “spring constant” associated with  $\omega_0$  ( $= \sqrt{k/m}$ ).

If  $x$  has the form  $x = Ae^{i\omega t}$ ,  $x$  will be:

$$x = (e/m)E/(\omega_0^2 - \omega^2) \text{ and}$$

$$P = -(e^2/m)n_e E /(\omega_0^2 - \omega^2)$$

## Appendix 1: Plasma or Natural Frequency of Electrons

- Using this simple model, one can calculate the polarizability of the material (see Appendix 1):

$$\kappa = 1 + 4\pi(P/E) = 1 + 4\pi (e^2/m)n_e [1/(\omega_o^2 - \omega^2)]$$

where  $n_e$  is the electron density,  $\omega$  the frequency of the x-rays, and  $\omega_o$  some natural frequency of the electrons in the solid, (in the Drude model its the frequency of the collective oscillations of the electron gas around the positive ion background or the so-called plasma frequency and equal to:

$$\omega_o = [4\pi n_e e^2/m]^{1/2} .$$

- For Si,  $n_e = 7 \times 10^{23} \text{ e/cm}^3$  and so the plasma frequency is:

$$\omega_o = 5 \times 10^{16}/\text{sec}$$

- For a 1 Å x-ray, the angular frequency,  $\omega (= [2\pi c/\lambda])$ , is  $2 \times 10^{19}/\text{sec} (>> \omega_o)$  and so we can write:

$$\kappa = 1 + 4\pi (e^2/m)n_e [1/(\omega_o^2 - \omega^2)] \approx 1 - 4\pi (e^2/m)n_e [1/(\omega^2)]$$

## Appendix 1: The Index of Refraction for X-rays

- The index of refraction,  $n$ , is just the square-root of the dielectric constant and can be written as:

$$n = \kappa^{1/2} = [1 - 4\pi((e^2/mc^2) c^2 n_e (\lambda/2\pi c)^2)]^{1/2}$$

$$n = \kappa^{1/2} = [1 - (n_e(e^2/mc^2) \lambda^2/\pi)]^{1/2} \approx 1 - (n_e r_e/2\pi)\lambda^2 = 1 - \delta$$

where  $r_e = (e^2/mc^2)$  is the classical radius of the electron ( $2.82 \times 10^{-13}$  cm).

- This simple model did not include any absorption of the incident radiation. A more detailed calculation would result in an expression:

$$n = 1 - \delta - i\beta$$

where  $\delta = (n_e r_e/2\pi)\lambda^2$  and  $\beta = \lambda\mu/4\pi$ , with  $\mu$  the linear absorption coefficient ( $I = I_0 e^{-\mu t}$ ).



## Appendix 2: Thermal Loading on Optics

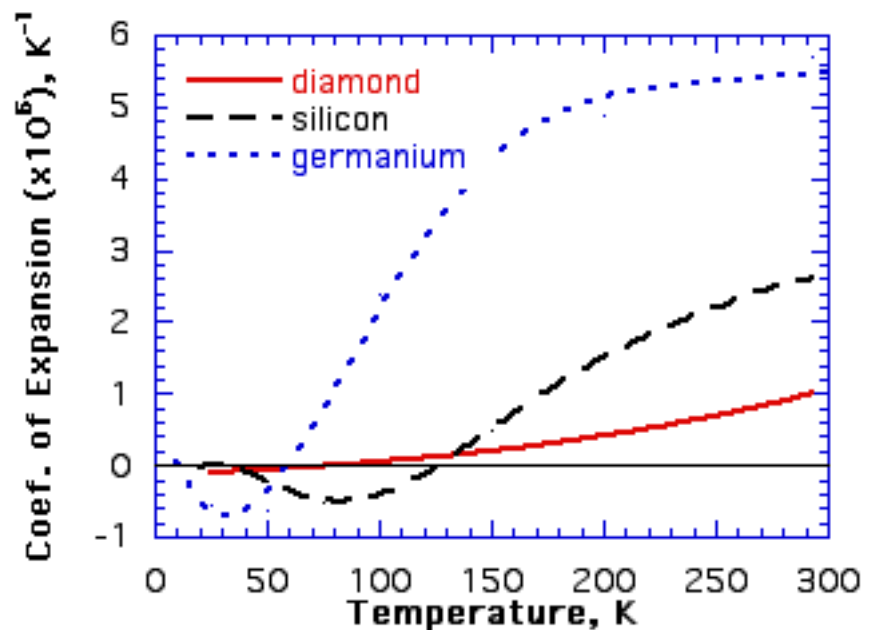
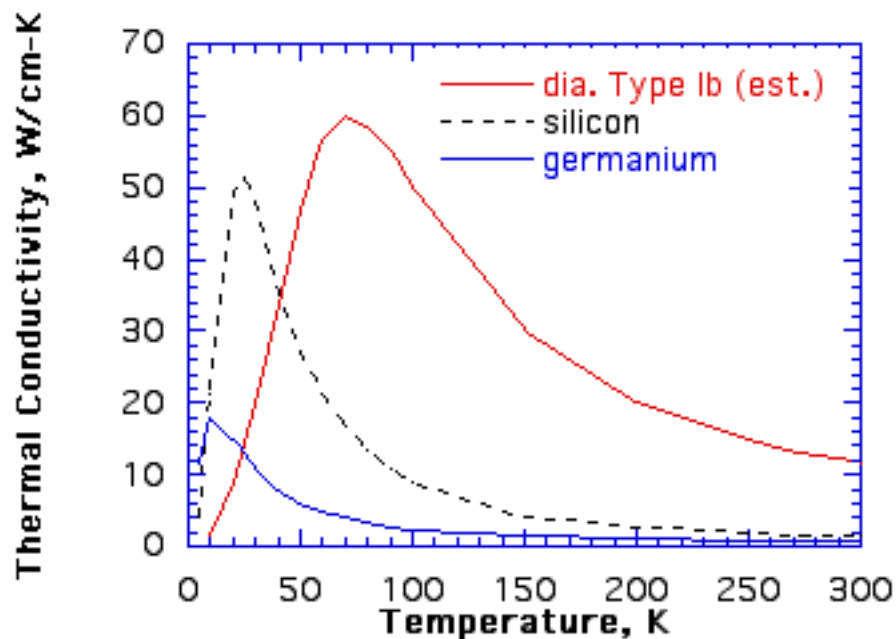
- Along with the enormous increase in x-ray beam brilliance from insertion devices comes unprecedented powers and power densities that must be effectively handled so that thermal distortions in optical components are minimized and the full beam brilliance can be delivered to the sample.

<u>Process</u>	<u>Approx. Heat Flux (W/mm<sup>2</sup>)</u>
Fission reactor cores	1 to 2
Interior of rocket nozzle	10
Commercial plasma jet	20
Sun's surface	60
Fusion reactor components	0.05 to 80
Meteor entry into atmosphere	100 to 500
<b>APS insertion devices (2.4 m and 100 mA)</b>	<b>10 to 160</b>

- In order to maintain the beam intensity and collimation (i.e., brilliance) through the optics, special attention must be paid to the issue of thermal management. (See Appendix 2 for details.)

## Appendix 2: Physical Properties of Si, Ge, and C(diamond)

- Thermal gradients,  $\Delta T$ , and coefficient of thermal expansion,  $\alpha$ , contribute to crystal distortions:
  - $\alpha \Delta T = \Delta d/d = \cot(\theta) \Delta\theta = \cot(\theta) \omega_D.$
- We therefore need to look for materials that have a very low coefficient of thermal expansion,  $\alpha$ , and/or have a very high thermal conductivity,  $k$ , so that the material cannot support large  $\Delta T$ 's.



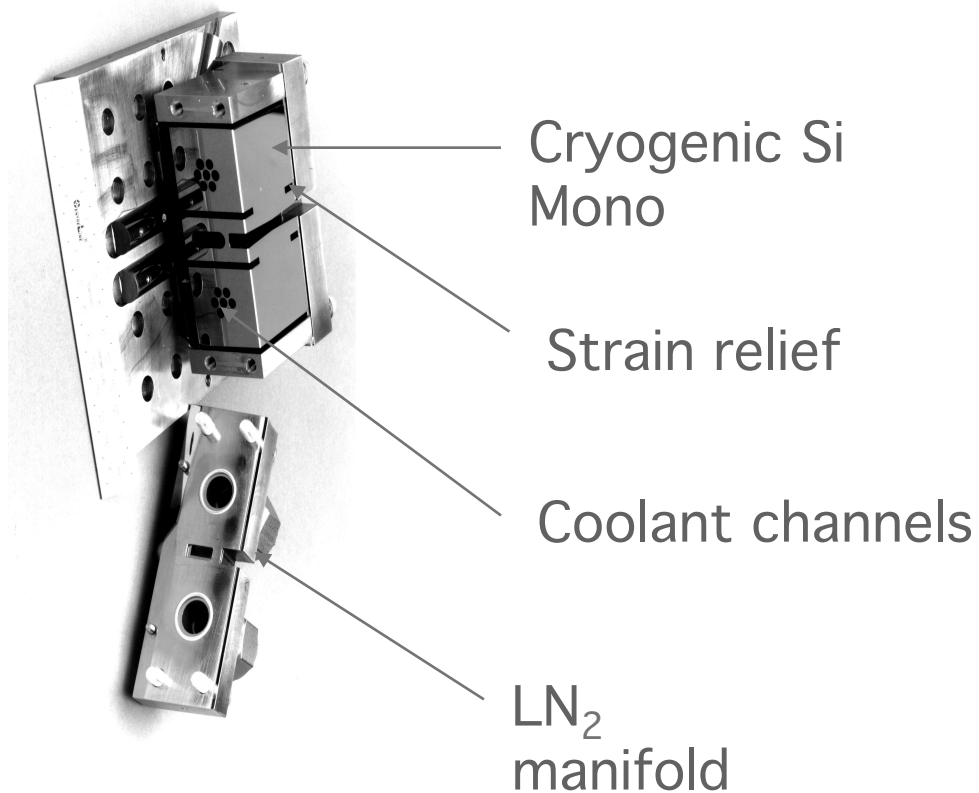
## Appendix 2: Figure of Merit (FOM) for Various Materials and Temperatures

- These conditions motivate us to use cryogenically cooled silicon or room temperature diamond as high heat load monochromators.

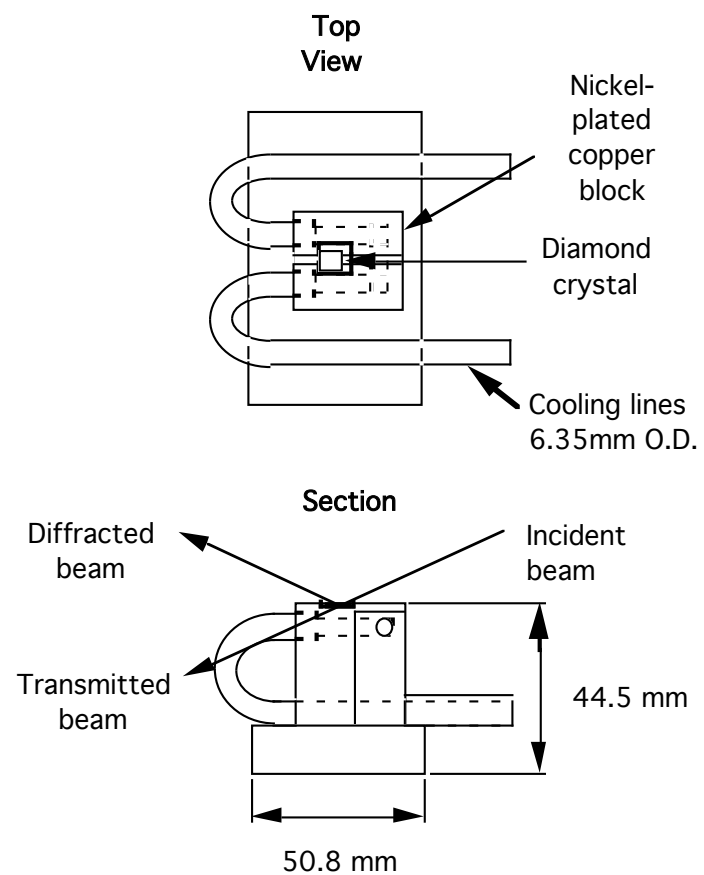
FOM of various materials

<u>material</u>	<u>k - thermal conductivity</u>	<u><math>\alpha</math> - coef. of thermal expansion</u>	<u>k/<math>\alpha</math> FOM</u>
Si (300°K)	1.2 W/cm-°C	$2.3 \times 10^{-6} / ^\circ\text{K}$	0.5
Si (78°K)	14 W/cm-°C	$-0.5 \times 10^{-6} / ^\circ\text{K}$	28
Dia. (300°K)	20 W/cm-°C	$0.8 \times 10^{-6} / ^\circ\text{K}$	25

## Appendix 2: Cooled Monochromators



Cryogenically Cooled Si Mono



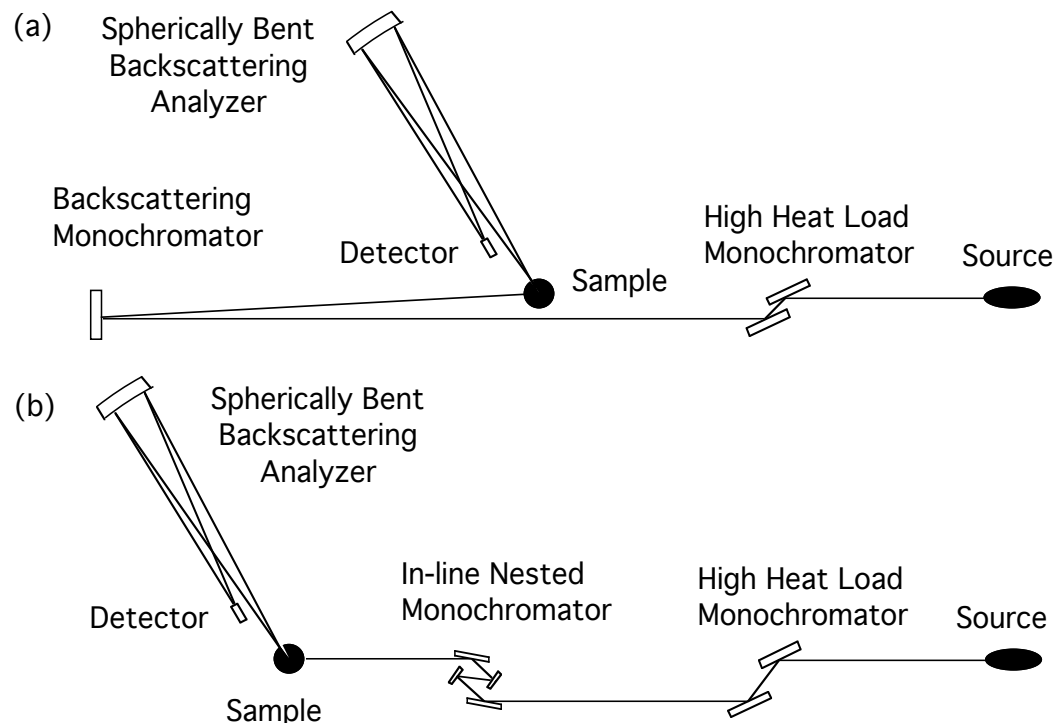
Water Cooled Diamond Mono

## Other Diffractive X-ray Optics - High Resolution Optics

- Recall that a Si (111) crystal has a energy resolution  $\Delta E/E \approx 10^{-4}$ , however milli-electron volt resolution monochromators and analyzers are often required for inelastic scattering.

$$\Delta E/E = \cot(\theta) \Delta \theta$$

as  $\theta$  approaches  $90^\circ$ ,  $\cot(\theta)$  goes to zero and the energy spread,  $\Delta E$ , gets very small and so a backscattering geometry is employed.



High-energy resolution (from eV to milli-eV) optics for inelastic x-ray scattering.

## High Energy-Resolution Optics (cont.)

- At  $\theta = 89^\circ$ ,  $\cot(\theta) = 1.7 \times 10^{-2}$ . For  $E = 20 \text{ keV}$  ( $0.64 \text{ \AA}$ ), then:

$$\Delta E = E \cot(\theta) \Delta\theta = (2 \times 10^4 \text{ keV})(1.7 \times 10^{-2})(10^{-5} \text{ rad}) = 3 \times 10^{-3} \text{ eV}.$$

- Note: For Si (111) at a Bragg angle of  $\theta = 89^\circ$ , the wavelength is  $6.2 \text{ \AA}$  (2 keV) and so to get near 20 keV at  $\theta = 89^\circ$ , we need to use a very high d-spacing such as Si (11 11 11).

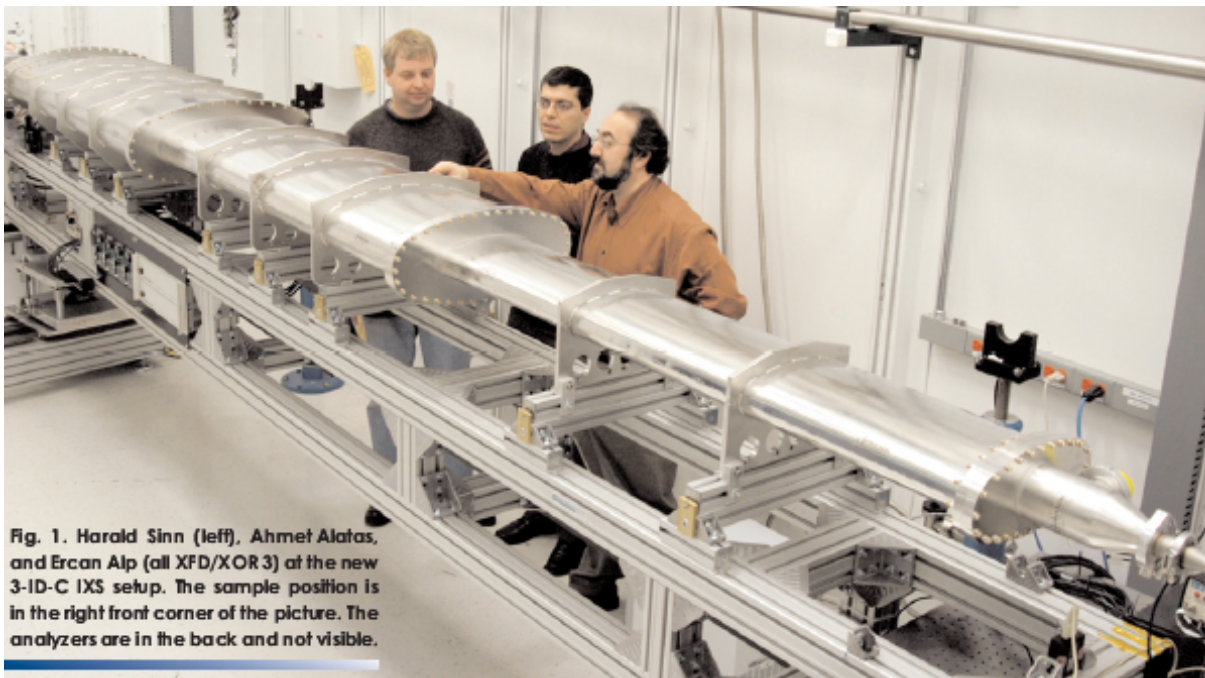


Fig. 1. Harold Sinn (left), Ahmet Alatas, and Ercan Alp (all XFD/XOR 3) at the new 3-ID-C IXS setup. The sample position is in the right front corner of the picture. The analyzers are in the back and not visible.

The inelastic scattering beamline at the APS.

